

# OPTICAL PROPERTIES OF MICROSTRUCTURED FIBRE TUNED BY FILLING WITH MAGNETIC LIQUIDS

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**Abstract.** *One of the key advantages of microstructured optical fibers is the multitude of ways to modify their properties inaccessible by conventional fibers. Outstanding alternative for adjust optical fiber light-guiding properties by bulk glass doping is provided by infiltration of optically active liquids into microstructured optical fiber, due to access directly to the light guiding core via air holes in microstructure. Following paper will report utilization of suspended-core, so-called high-delta, microstructured optical fiber as an optimal medium for tuning and developing optical devices using infiltration technique. Means of filling such fiber with variety of liquids will be presented and guided light affection in bare silica glass fiber will be demonstrated.*

## Keywords

*High delta fibers, microstructure filling, microstructured optical fibers, suspended core fibers.*

## 1. Introduction

Progress in fiber optics manufacturing technology gave us access to the broad spectrum of variety new profiles tailored for their specific applications. Among others, microstructured fibers became commercially available in many different designs as they are evolving and perfecting very rapidly. One of those refined designs is suspended-core microstructured optical fiber (MOF). In suspended-core (so-called high-delta) fibers we are looking for highest possible difference of refractive indices between core and cladding region, which brings certain unique properties [1]. Natural solution to satisfy such conditions are microstructured fibers because of their native low cladding refractive index, defined by

their ratio of air and solid phase in microstructure (filling fraction, FF). For suspended-core MOF it means to create narrowest possible bridges between air holes in microstructure, e.g. to approximate FF very close to 1.

Consequently, perfect suspended-core MOF act as light-guiding strain surrounded by air. However, due to silica melt inconvenient ration of surface tension and viscosity is quite difficult to fulfill this presumption. Resulting fiber made out of silica glass then use to have quite thick bridges between holes. This lowers FF and allowing light leakage from the core region, which is pure loss in result. Nevertheless, such fiber is commercially available as custom fiber from NKT Photonics [2] and can be used for experiments as well for potential applications. Interaction among guided light and holes filling in such microstructured fibers was already proved [3], [4]. As for the filling, variety of liquids can be used with basically no limitations, as surface of microstructure holes can be treated to resist volatiles. Furthermore, recent progress in technology allows us to use not only conventional solutions, but also colloidal solutions of micro- and nanoparticles, liquid crystals and polymers.

Main target of the whole experiment was to demonstrate interaction among the light guided in the fiber core and liquid filled inside of the holes surrounding the fiber core. This approach can provide means to drive fiber optical properties, as losses or band gap, with external quantities which generally does not affect light guiding mechanisms, e.g. magnetic or electric field.

## 2. Experiment Preparation

The liquid chosen for the experiment is particularly unique. It is a colloidal solution of sub-micron su-

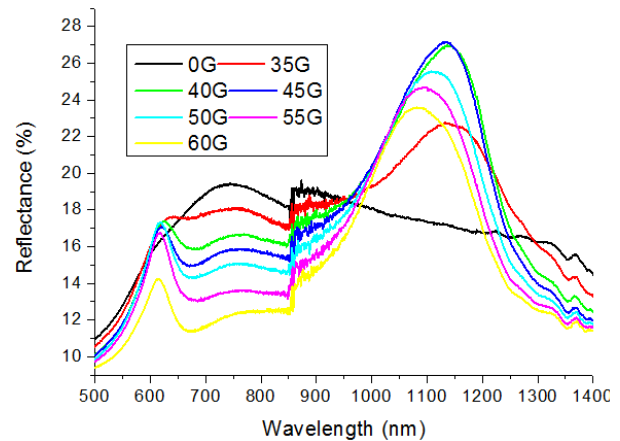
perparamagnetic microparticles. Such colloid has very specific behavior and it can provide some interesting properties. One of the most attractive in the field of optics and photonics is the ability of forming photonic band gap in the presence of magnetic field. Particles randomly dispersed in solution exhibits Brownian motion in normal conditions; however, under the presence of magnetic field they align to chains accordingly to magnetic forcelines, forming well-defined periodic columnar structure of sub-micron dimensions. Such structures are well-known in photonics as photonic crystals; structures possibly exhibiting reflectance bands for certain light wavelengths. Proper choice of particles size affects distances between chains and consequently optical properties of formed crystal [5]. The experiment itself is designed to give a better view to the properties of liquid filled fiber and to possibilities of tuning the light propagating through such fiber. However, filling procedure itself bring certain difficulties, which affecting experiment reproducibility and results accuracy and hereafter described experiment is just first step in utilizing and exploring whole new field, where microstructured fibers can be exploited.

## 2.1. Microparticles

For the experiment was chosen commercially available aqueous solution of 300 nm diameter polystyrene (PS) microparticles (concentration  $25 \cdot 10^{-3} \text{ kg} \cdot \text{l}^{-1}$ ). Particles contain 54 % of weight of superparamagnetic iron oxide nanocrystals homogeneously incorporated in polystyrene sphere, made by Particles GmbH [6]. This technology allows preserving superparamagnetic properties of nanocrystals in order of magnitude bigger particles [7].

At the initial stage was also measured refractive index of microparticles solution by two methods, Abbe's refractometer and using fiber long period gratings [8]. However measured values match the value of solvent itself (water) with  $10^{-3}$  accuracy. This may be caused by relatively low fraction of microparticles in solution (approximately  $25 \text{ g} \cdot \text{l}^{-1}$ ).

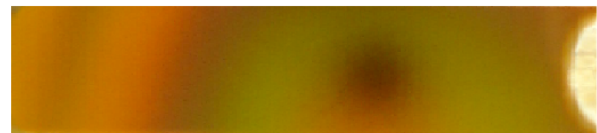
Spectral characteristics of solution were measured by spectrophotometer Perkin-Elmer (Fig. 1). These results are particularly important to find magnetic field strength at which particles are stable in solution, as the spectrophotometer scanning cycle takes up to five minutes. In this particular case, optimum magnetic field strength lies between 45 and 50 Gauss, where particles form structure stable in time. For higher values of field, magnetic force is too strong and particles tend to precipitate. In the case of lower magnetic field, particles are affected more with other forces and formed structure tends to blur.



(a) Spectral characteristics of used microparticles in various magnetic fields.



(b) Solution in cuvette without the presence of magnetic field.

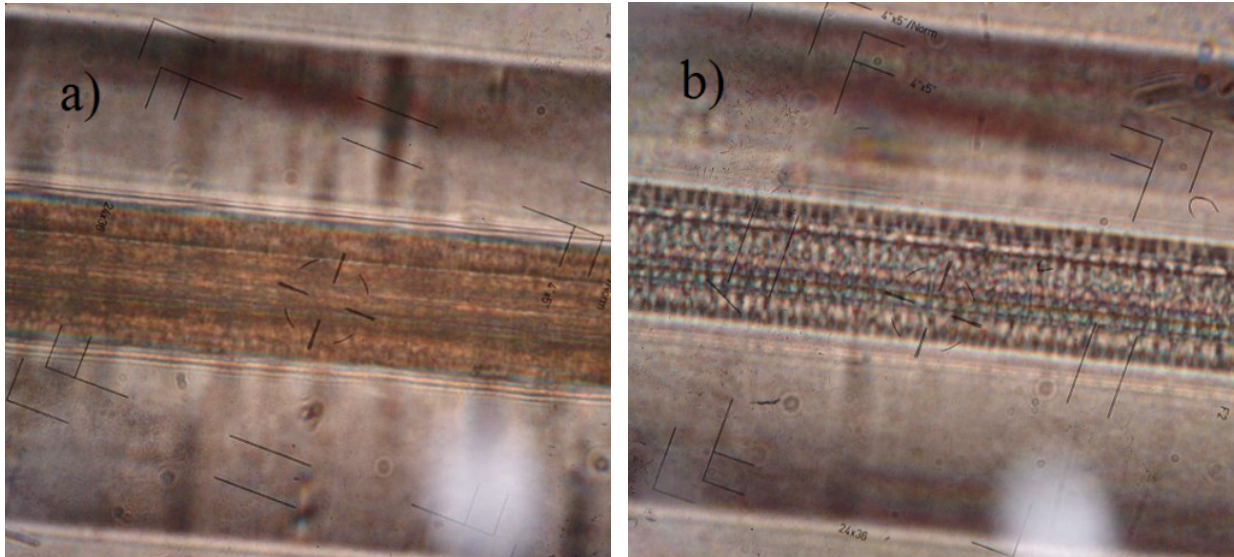


(c) Solution in cuvette affected by the magnetic field of bar-type magnet.

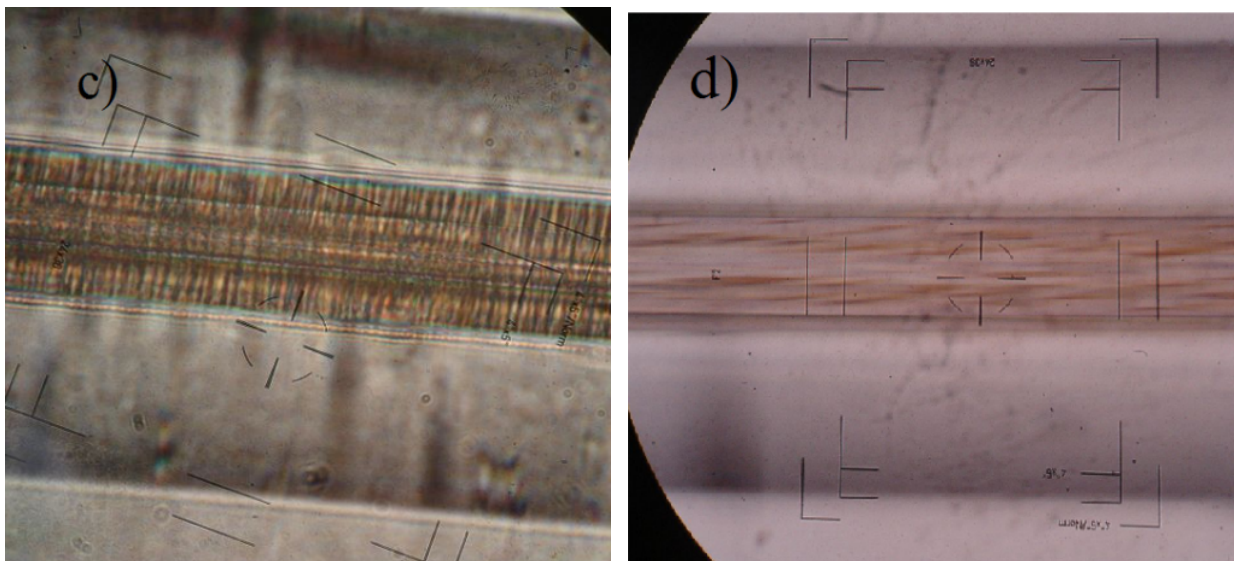
**Fig. 1:** Reference reflectance measurement (100 %) was taken from spectrophotometer build-in integration sphere, in which was then placed cuvette with solution.

As could be seen from the Fig. 1(a), reflectance is quite constant for the NIR and dropping for the visible. This corresponds with unorganized, Brownian motion of particles in solution creating brownish, opaque liquid within cuvette (Fig. 1(b)). A remarkable change occurs in the presence of magnetic field, particularly for the range of magnetic flux density 35–60 Gauss. Significant reflectance band appears in NIR with a maximum at 1150 nm for the magnetic flux density around 45 Gauss. Another smaller peak appears around wavelength 600 nm. This can be observed as noticeable color change of the region affected by the magnetic field (Fig. 1(c)).

Microscopy images (Fig. 2) shows the details of particles in the solution with and without the presence of magnetic field. From the Fig. 2(b) can be seen regular arrangement of the columns formed by particles clustering in the presence of magnetic field (magnetic force lines direction is parallel with view direction). Fig. 2(c) and Fig. 2(d) shows the view from the sides (magnetic force lines direction is perpendicular to the view direction).



(a) Solution of particles inside the fiber microstructure capillaries without acting magnetic field. (b) Solution of particles inside the fiber microstructure capillaries with the magnetic field acting parallel with view direction.



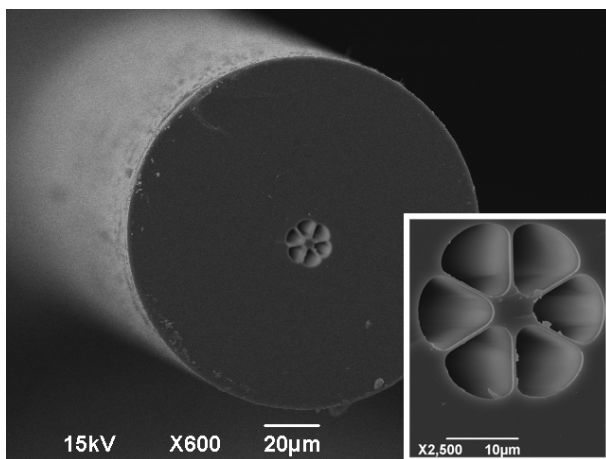
(c) Solution of particles inside the fiber microstructure capillaries with the magnetic field acting perpendicular to the view direction. (d) Solution of particles inside the fiber microstructure capillaries with the magnetic field acting perpendicular to the view direction.

**Fig. 2:** Solution of particles inside the fiber microstructure capillaries.

## 2.2. Fiber

As mentioned above, fiber with a small core and high air filling fraction suitable for filling experiments is commercially available (Fig. 3). Strong advantages of commercial fiber are regular dimensions allowing using common components and procedures for conventional fibers, as connectors, cleaving, etc.

Necessary technique for successful conducting experiments is splicing with conventional fibers, required to ensure stable and reproducible launching and detecting conditions. Process of splicing such specialty fibers is more difficult than in conventional fibers case and factors affecting good quality splices of microstructured fibers with conventional describes Murawski et. al. in [9].



**Fig. 3:** SEM image of microstructured fiber used for experiments. Core region is approximately 5  $\mu\text{m}$ , microstructure 30  $\mu\text{m}$  and fiber 125  $\mu\text{m}$  in diameter.

## 3. Experimental

For the experiment was necessary to develop simple and stable setup, as dealing with fiber optics is very sensitive on launching and positional conditions, and inclusion of liquids in the whole system does not improve the stability at all. Goal of the experiment was to stabilize colloid inside the fiber and reach reproducible modulation of light guided in the fiber core by switching colloid from scattered sol into organized rod-like crystal structure with magnet.

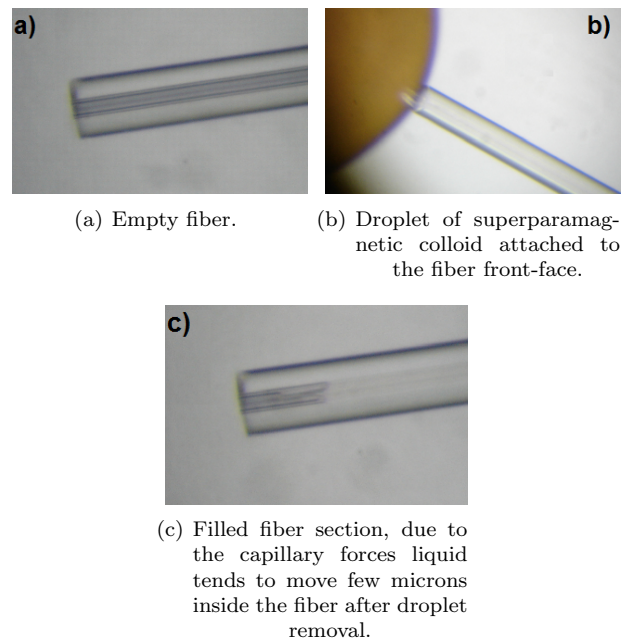
Experiment itself was divided into several steps, where stable input/output conditions in microstructure fiber was solved, liquid infiltration and mobilization within fiber capillaries realized and experimental setup with magnet constructed.

To affect particles infiltrated inside of the fiber, two magnets were used. First, standard bar-type magnet

together with non-magnetic micrometric mount and magnetometer for initial experiments to check the response of the solution was utilized. In the next step, especially for fast switching of magnetic field and precise magnetic field strength control, bar-type electromagnet was used. Measurement of field strength also suggests, that for the very small size of fiber capillaries magnetic field can be considered as homogeneous.

### 3.1. Infiltration

Essential for experiments was to find the way for infiltrate colloid into the desired section fiber and that immobilize it. Infiltration of water-based liquids appears to be relatively easy, as due capillary forces water-based enters fiber microstructure by themselves immediately after immersing fiber end-face into solution. To keep homogeneous infiltration inside all microstructure holes, high precision cleaving of fiber is necessary. Homogeneity of infiltration should be easily checked with microscope, as well as length of infiltrated region (see Fig. 4).



**Fig. 4:** Infiltration of microstructured fiber (10 $\times$  microscope images).

Of course, after such infiltration, colloid is very sensitive on outside effects; it tends to dry, run out of the fiber, can be contaminated etc. It is necessary to move colloid further to the fiber and seal fiber ends without losing optical linkage with filled region. For sealing is optimal fiber splicing, where can be used industrial fusion splicer after certain experience. For colloid mobilization and stabilization in desired microstructure section was developed technique utilizing ferrofluid and photopolymer.

Before superparamagnetic colloid infiltration, ferrofluid was introduced to the other end of microstructured fiber and with magnet guided further. Because of colloid can infiltrate capillaries even if the other end of the capillary is sealed, infiltration process is not corrupted. After colloid infiltration, ferrofluid is guided with the magnet back to the other end of the fiber. Vacuum makes colloid to move and by this way is possible to position it to the desired region. During the process, small droplet of photopolymer is infiltrated after moving colloid. At desired position is photopolymer solidified with UV light and colloid is immobilized inside of the fiber. Thereafter, contaminated fiber can be cleaved and fiber is ready to splice with launching and sensing pigtailed. Schematically, resulting filled section is on Fig. 5.

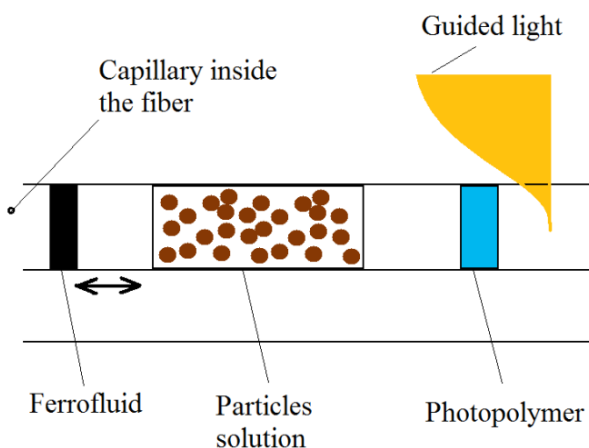


Fig. 5: Visualization of microstructured fiber capillary filled with immobilized colloid.

### 3.2. Experimental Setup

For experiments, two different measurements were used, backreflection and transmission respectively. However, due to relative simple setup, which was achieved by maximum integration of the optical path it was possible to conduct both measurements on the same components without modifying the setup.

As was mentioned above, to receive stable and reproducible response from filled microstructure fiber is necessary to splice it with conventional fibre. Considering core size of used microstructured fibre, appropriate conventional fiber of similar core and mode field diameter has been chosen. Thus, by choosing appropriate splicing conditions to prevent microbending in joint, NA mismatch became dominate in splice losses and reproducibility of setup with splice ensured.

Figure 6 is showing the setup of the experiment used after finalizing filling and splicing microstructured sample. Filled microstructured fiber was after splicing with

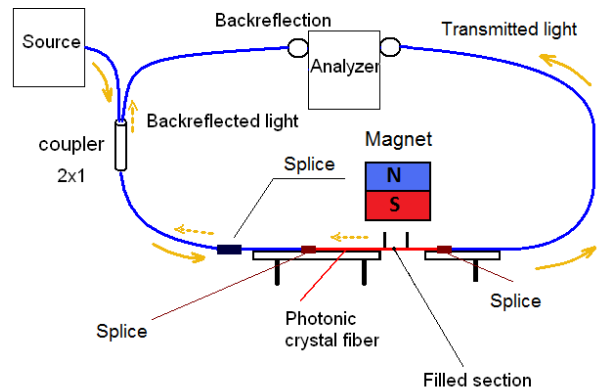


Fig. 6: Schematic setup for filled fiber experiments for backreflection and transmission measurements.

conventional fibers fixed on brass V-grooves with access to the filled section. As the source was used single-modulated laser diode pumped  $\text{Yb}^{3+}$  fiber optic amplifier at central wavelength of 1060 nm. Light was launched into the experimental fiber thru laboratory grade 50:50 coupler, which was used to guide backreflected light to the spectral analyzer. Other available channel of analyzer was used to monitor transmission output of the fiber.

### 3.3. Measurement

In the first step, both transmission and backreflection signals were measured without magnetic field affecting the fiber. This background signal was used to check the stability of the setup and to set the background signal for zero magnetic field configurations. Thereafter, direct magnetic field was applied and the signal was measured again. As could be seen from graphs, both in backreflection and transmission has a magnetic field notable influence on signal intensity.

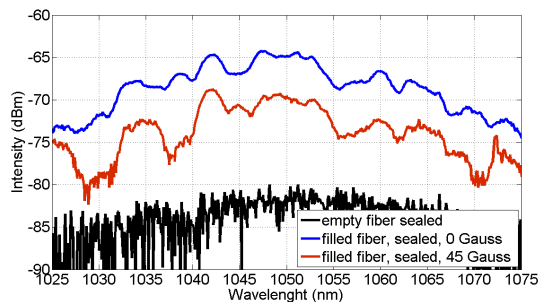
For experimental purposes, magnetic field was applied both parallel with light propagation direction and perpendicular. However, intensity of the signal is close to the threshold level of the spectrum analyzer so that the resulting curve is quite noisy. To reach readable signal level, reflections from fibers ends and splices had to be suppressed enough.

## 4. Results and Discussion

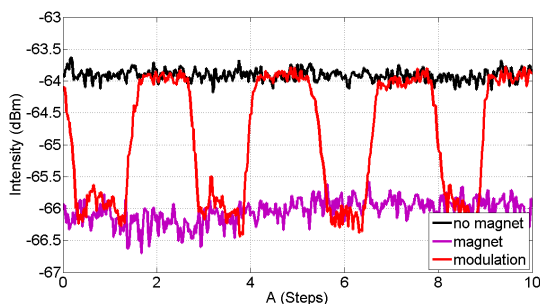
Although signal levels were very low and therefore affected by many influences arising from the splices, connection and housing of the tested fibers, setup provided stable and reproducible effect on the output. As can be seen on the Fig. 7 and Fig. 8, signal levels exhibits notable difference in the intensity level for situations, where infiltrated colloidal solution was in dis-

persed state and situations of organized state in the presence of the magnetic field.

As was expected, magnetic field applied parallel with light propagation had no effect on the guiding properties of the fiber. Photonic structure formed in the solution was present only in the non-guiding holes along the fiber core, thus guided light in the core cannot be affected by this effect. More interesting were the results from experiment with the magnetic field applied perpendicular to the light propagation. As the particles aggregates into columns oriented in the direction of magnetic field, high-index rods touching the core surface are formed in the fiber cladding.



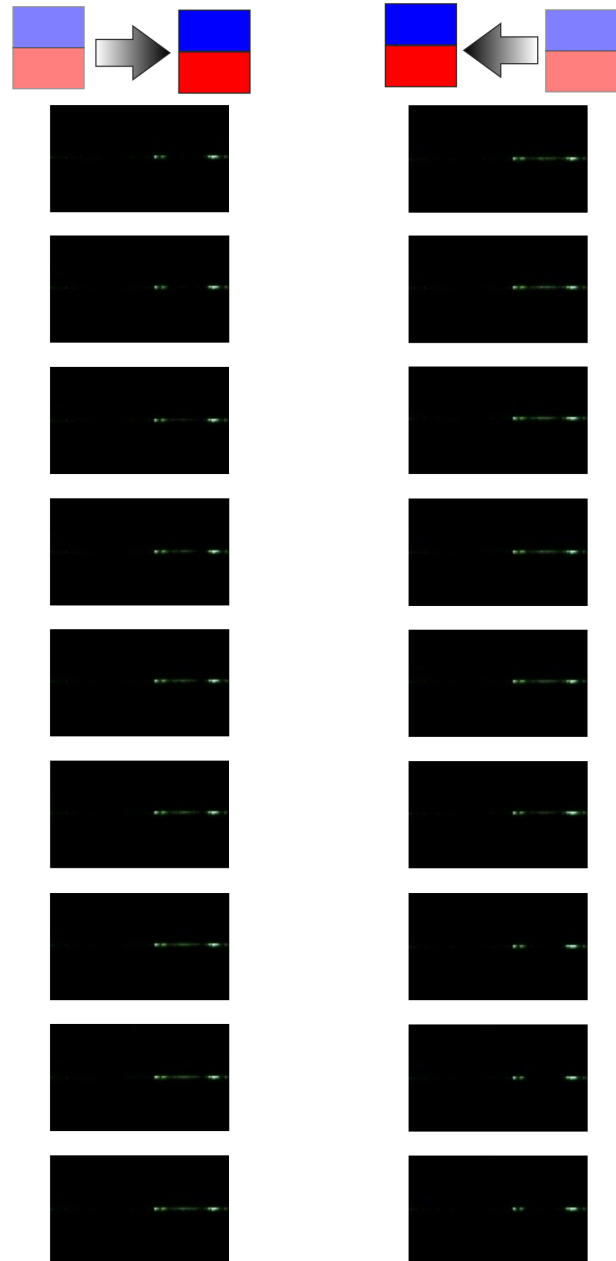
**Fig. 7:** Measured backreflection in the setup as on Fig. 6. Black curve is the background signal for the case of setup with empty fiber sealed with photopolymer. Red curve is the backreflection signal from sealed fiber filled with colloidal solution without influence of magnetic field. Blue curve is the backreflection signal from sealed fiber filled with colloidal solution in the presence of magnetic field 45 Gauss.



**Fig. 8:** Transmitted signal track in time at wavelength 1060 nm. Response is for magnetic field 0 (blue line) and 45 (magenta line) Gauss and for magnetic field switching from 0 to 45 Gauss with approximately one second period (red line).

Signal is exhibiting significant decrease in intensity in the presence of magnetic field, both in backreflection and transmittance, and this effect is wavelength independent in the tested range of 540–1550 nm. This corresponds with observation of filled fiber with a camera, where notable scattering and “leaking” of light from filled section in the presence of magnetic field was observable. During experiment, variety of fiber lengths were infiltrated in the range from 5–15 mm, but no

significant change in the experimental setup response was observed. We assume that the majority of the observed effect occurred in the first few millimeters of infiltrated section.



**Fig. 9:** Images of light scattered in filled fiber section in the presence of a magnet. Filled section is approximately 10 mm long, light was launched from the left. Magnet was moved in constant distance from fiber along filled section in the direction of passing light and back.

Simple filling technique however allows infiltrating only section in the mentioned range; sections shorter than 5 mm showed significant inhomogeneity, where solution was stabilized in a different position within every hole of the microstructure. On the other hand, infiltration of larger than 15 mm section was too difficult to mobilize. Considering the controllability of infiltrated

solution, optimum length of infiltrated section appears to be about 10 mm (Fig. 9).

Used configuration did not allow exact measurement of scattered light leaking from the fiber, signal loss is however equivalent both in transmittance and back-reflection, approximately at the level of 3 dB. Signal spectral shape was not affected and no band gap was observed. It is assumed, that interaction among light guided in the fiber core and colloid in an organized state (in the presence of magnetic field) was realized by deconfinement of part of the light from the fiber core due to altering the conditions on the hole-core interface.

When the conditions in the cladding region (holes) was changed, part of the light propagating in the evanescent field was due to the high-refractive index rods, formed by microparticles in the presence of magnetic field, deconfined and scattered to the surrounding media.

## 5. Conclusion

In paper was presented an effective method to control light, guided in microstructured optical fiber, by external quantities using functional filling within microstructure. Practical way of filling, immobilization and activation of magnetic liquid within microstructure was demonstrated and transmission characteristics of microstructured optical fiber were reversibly changed. Such method can help in development of both fiber optics technologies, opening new fields of their utilization and optically active materials in the other hand, well defining the needs and requirements of fiber optic based devices.

There is a number of possible ways for further exploitation of presented technique; example can be development novel types of optical fiber with active cladding for fiber lasers, where active media can be introduced into the fiber microstructure after the manufacturing process, broadening list of possible materials and allowing their alteration in short fiber sections. Further applications suggests for example hollow-core PBG fibers, where the air-guiding core can be filled with microparticles or nanoparticles solutions, allowing direct control of guided light.

Presented method can improve utilization of microstructured optical fibers in the field of active optical components for a variety of application, as it allows tuning optical properties of fiber without intervening into fiber material or structure. Cheap, mass produced microstructured fiber can be used this way in different areas and for different purposes, based on substance presented within microstructure.

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