Ferrofluidic sensitive element based on miscrostructered fiber

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This article discusses problems related to the utilization of ferrofluid, infiltrated in a microstructured optical fiber and controlled by an external magnetic field. It summarizes the results obtained by the authors with the fiber-filled with this substance and subjected to harmonic or impulse field. It is shown that significant magneto-optical effects are observed here that are promising for designing tunable fiber elements.

Keywords: Fiber optics; ferrofluid; magnetic fluid; magnetic control.

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1. Introduction

There are many ideas and approaches related to how to control the light propagating through the optical fiber by changing the characteristics of the medium surrounding its core. Here we will focus on magnetic phenomena: first, this is an important physical problem itself, and, it can open a serious prospect for creating magnetically controlled fiber optoelectronic devices of a new type. As an active substance, we will consider a ferrofluid (FF) or, a magnetic liquid, which is a very convenient object to handle, since it is rather easy to fill the capillaries of a microstructure with it. For example, one can surround the central core of the fiber with the transmitted light. FF itself has been intensively studied for a long time as a unique system that combines magnetic properties with fluidity [1]. In the last decade, the interest has been increased due to the new proposals that have appeared for the FF application in basic research [2-4], biomedicine [5-6], and also, what is important for us, in photonic devices [7-8]. The number and variety of the latter applications are quite large, here we will give some illustrative examples only. These include: optical switches [9], light modulators [10], controlled diffraction gratings [11-13], and different types of sensors [14-16].

The principle of operation of these devices was based on a physical phenomenon that a magnetic field applied to FF changes its optical characteristics, including the refractive index n. The mechanisms of these changes in the material parameters are mainly associated with the formation of structures-agglomerates composed of nanoparticles of the solid phase of the substance. The agglomerates' size and their growth dynamics are governed by the field [17]. There are, probably, other contributions to this effect determined by the properties of individual nanoparticles [18]. We would like to emphasize that, despite a significant number of previous studies, the detailed nature of such phenomena cannot be considered as completely understood. Most likely, they are complex; however, if at least some part of the magneto-optical response is determined by the rapid processes, it is possible to control the FF-based element at a high frequency.

Below, we present results on microstructured fibers filled by FF, with various versions of external influence.

2. Ferro fluid

FFs are the special colloids with the solid phase consisted of particles of a magnetic material, having diameter d of the order of ten nanometers. Magnetic dielectrics (ferrites) and, sometimes, metals are used as such substances. The adhesion of the nanoparticles is prevented by special synthesis of FF performed in such a way that every particle is covered with a shell, formed by surfactant molecules, or (for some liquid phases) by a double electric layer.

The solvents can be hydrocarbons, water, oils, etc. The combination of various liquid media with a variety of solid magnetically ordered materials generates a lot of FFs. For the applications, described above, the best is the colloidal solution of magnetite (Fe_3O_4) in kerosene. Water is poorly suitable for infrared applications; oils are too viscous and introduce high losses. The solution based on kerosene has an acceptable transmission in the infrared range, and its refractive index can be selected by varying the concentration of magnetite particles. This is done to satisfy total internal reflection conditions at a boundary of the fiber core and the filled microstructured cladding. The transmission of the light is controlled by applying an external magnetic field that changes n. It should be noted that FF refractive index also depends on light intensity [19] in the high power regimes. However, at moderate power levels (of a few mW-scale at a wavelength of 1550 nm) the nonlinear optical effects are negligible.

We used the FF, obtained by the hydrolysis of ferric chloride and sulfate ammonia with the extraction of magnetite particles from the reaction mixture with a kerosene solution of oleic acid, which forms a surfactant layer at the surface of the Fe_3O_4 particles. According to [20], the particle sizes of the material produced by this technique have log-normal distribution (normal distribution of the logarithm of the variable) with the average median value of $d = 8.2$ nm and the scaling parameter (the characteristic parameter of distribution) 0.17. For these values and the anisotropy constant is known for

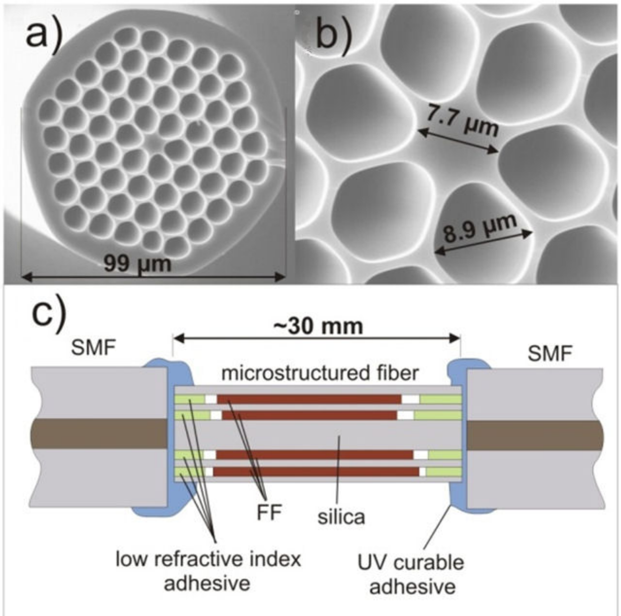

FIGURE 1. Magnetically controlled element based on microstructured fiber: a) the view of fiber from the end, b) the size of the capillaries and the central core, c) connection of the structure with single-mode fibers.

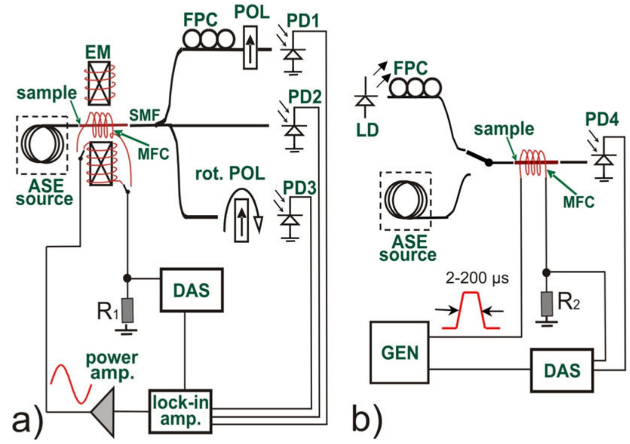

FIGURE 2. Block diagrams of experimental setups with a magnetically controlled element embedded in an optical fiber: a) The configuration with a sinusoidal-wave modulated magnetic field, and b) That with the pulsed magnetic field. ASE source - amplified spontaneous emission source; MFC - magnetic field coil; FPC - fiber polarization controller; POL - polarizer; PD - photodiode; DAS - data acquisition system; EM - electromagnet; GEN - pulse generator; LD - light-emitting diode.

magnetite, the time of orientation jumps of the particle's magnetic moment can be estimated to be less than 10^{-7} s [1] that is less than the time of rotation of the particle as a whole. The volume concentration of magnetite satisfying the waveguide formation condition in the microstructured fiber was 0.25 %.

3. Optical fiber

As a basis for fabrication of the magnetically controlled element, we used a segment of the silica microstructured optical fiber, the view of which, with important dimensions, is shown in Fig. 1. Scanning electron microscope images of the fiber cross-section are shown in Figs. 1a) and b), the design of the device is demonstrated in Fig. 1c). One can see that this system has a central core, surrounded by a set of capillaries (that imitate its outer cladding), filled with FF. The length of the segment was about 3 cm. The geometric sizes of this element and the parameters of its constituent media were selected to provide conditions for the total internal reflection at the light wavelength $\lambda = 1.55$ μm. The overlap of the fundamental mode with the FF material for the selected magnetite concentration was estimated at 7%. On both sides, the structure was connected to single-mode fibers (SMF) with a mode field diameter of 10.4 μm using a special low refractive index adhesive.

4. Experimental setup and results

Experimental setups used for our magneto-optic measurements are shown in Fig. 2 with explanations of the abbreviations given in the figure caption. Figure 2a) illustrates the case of the externally applied sinusoidal field, which was created either by the coreless coil MFC or by EM with the ferrite core. These ensured longitudinal or transverse orientation of the magnetic field to the fiber axis respectively. Figure 2b) demonstrates the circuit utilized for experiments with the pulsed longitudinal magnetic field. The physical mechanism of modulation was, as it has been mentioned above, the field-induced change in the refractive index n of the FF, *i.e.* changing conditions of the light propagation along with the fiber core.

In the configuration presented in Fig. 2a, the sample was exposed to the broad-band optical radiation from the non-polarized erbium doped fiber amplified spontaneous emission (ASE) source ($\lambda = 1535 - 1571$ nm) via SMF. The light passed through the sample was detected by PDs, and its polarization properties were evaluated using rotatable analyzer (rot. POL) or by conventional three-loop fiber polarization controller (FCP) followed by fiber analyzer. The measurements were performed with the harmonic magnetic field $H(t) = H_0 \sin(2\pi f + \varphi)$ in the frequency range of $0.15 - 20$ kHz. The magnetic field amplitude could be varied within $0 - 250$ Oe. The maximum modulation contrast of the output light achieved in the experiments was about 0.6%.

In the configuration shown in Fig. 2b, the laser diode LD ($\lambda = 1532$ nm) or the same ASE source were used as the light sources. The longitudinal magnetic field was created by the low inductance MFC powered by the pulse generator GEN. This ensured the square-shaped pulses in the coil with durations from 2 to 200 μs and resulted in the magnetic field amplitude up to 2 kOe. When using a polarized source, the polarization of light at the fiber input was set by FPC.

Similar methods were utilized in papers [21,22], where a more detailed description of the schemes and features of their

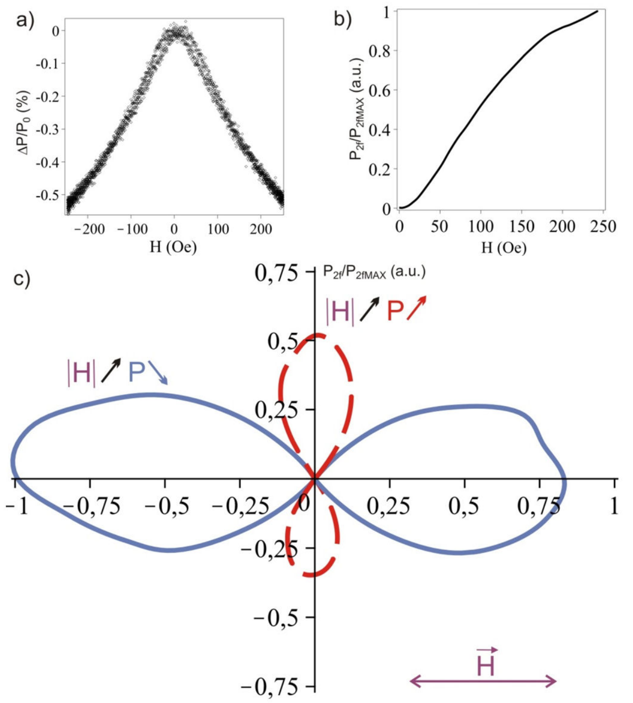

FIGURE 3. The results of experiments with harmonic modulation of the transverse magnetic field ($H_0 = 25$ Oe, $f = 150$ kHz). a) Modulation depth, corresponding to the maximum positive and negative values of the alternating field H, b) field behavior of the normalized amplitude of the second harmonic, c) angular dependence of the response at the second harmonic $2f$ for different mutual orientations of the magnetic field and the light polarization.

operation can be found. Below we present our most significant experimental results obtained with these configurations.

Examples corresponding to the case of sinusoidal modulation are presented in Figs. 3 and 4. In the transverse geometry, when the total transmitted power P was detected without using polarizers, the response was observed at even harmonics of the modulation frequency f (even field effect), the results are shown in Fig. 3a and 3b. Figure 3a presents the depth of modulation as a function of the field, in Fig. 3b the field dependence of the amplitude of the second harmonic of the response is demonstrated. Using the polarizer in a rotation holder, it was also possible to detect the polarization features of the signal (Fig. 3c).

The action of the longitudinal sine-wave magnetic field has also been investigated. The output signal observed in the absence of polarizers at the even harmonics did not decrease with increasing H, as in the previous case, but increased instead. The $P_{2f}(H)$ function remained symmetric and reached a maximum value of 0.3% at $H_0 = 250$ Oe. The first harmonic response was recorded using FCP at the input and the polarizer at the output of the microstructured fiber. In this configuration, it was possible to suppress the signal (Fig. 4a) or to obtain it in-phase (Fig. 4b), or in antiphase (Fig. 4c) with the applied modulated magnetic field. The maximum modulation depth observed here was about 1.5%.

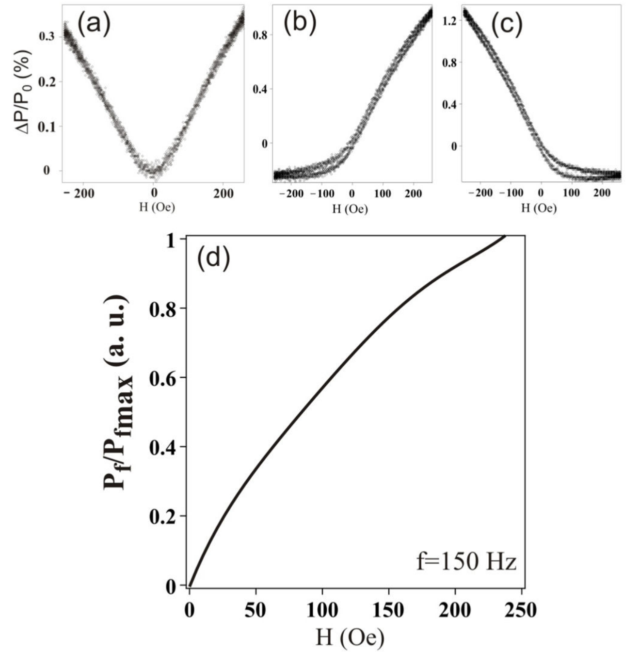

FIGURE 4. Influence of the longitudinal sinusoidal field on the first harmonic response: a) suppressed signal; b) signal in phase with the periodic field; c) signal in antiphase with the field, d) the normalized magnitude of the response versus the field amplitude.

A typical dependence of the normalized signal power at the first harmonic on the applied field is shown in Fig. 4c.

Note that the cut-off frequency of the sinusoidally modulated signal was of the order of several kilohertz. There was, however, one exception: the response recorded at the first harmonic in the longitudinal geometry turned out to be somewhat high-frequency. Still, we must conclude that the main processes that determine the response in the continuous-wave field mode are rather slow.

Pulsed experiments realized using the setup shown in Fig. 2b demonstrate the dynamics of the magnetic system and its underlying properties including the presence of a weakly expressed fast component. Figure 5 shows the forms of the acting magnetic field and the optical response under various conditions (see figure captions). The output signal obtained with non-polarized light was an even field effect, *i.e.* it did not change when the field direction changed. It had quite a long rise and fall times, which is shown in Fig. 5a and 5b for different pulse durations.

With the circular polarization of the incident light, the response profiles obtained at different field orientations differed sharply one from the other (Fig. 5c). The sum of these functions turned out to be close to the even signal observed for the unpolarized light. Their subtraction resulted in an odd component with a much faster rise, following the shape of the pulse leading front (with the characteristic time estimated as ≈ 0.2 μs). Thus, this response revealed components that are determined by some different physical mechanisms.

According to [22], they are, probably, associated with the rotation of the magnetic moments inside the nanoparticles and with the rotation of the nanoparticles as a whole. The

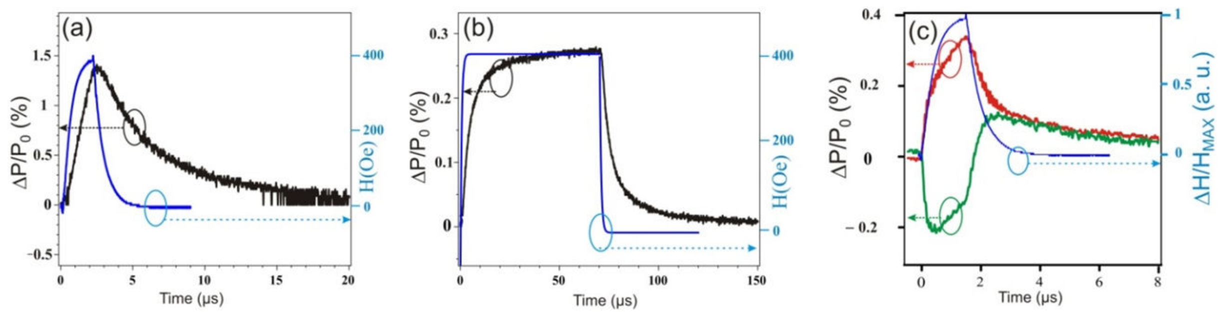

FIGURE 5. Pulse field response: a) unpolarized input light, pulse duration is 2 μs (blue-pulse, black-response); b) unpolarized input light, pulse duration is 70 μs (blue-pulse, black-response); c) input light with circular polarization, pulse duration is 2 μs (blue-pulse, red and green-responses for the opposite directions of the magnetic field).

important thing here is the presence of a fast response component, which allows us to hope for the possibility of implementation of a high-frequency modulation using FF.

5. Conclusion

Summarizing, we have presented experimental data related to the magnetically controllable photonic elements based on ferrofluid, which, in our opinion, are of interest both from a fundamental physical and practical point of view. The direct utilization of such a device for optical signal modulation can be limited because of the relatively low speed of the processes that determine the reaction of the ferrofluid to a magnetic field. However, such a device can be useful for developing various types of detectors, for example, the magnetic field probes. Besides the presence of the fast component of the magneto-optical response still allows us to expect that in the future the frequency range of ferrofluid-based elements can be expanded.

Acknowledgments

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1. C. Scherer and A. M. Figueiredo Neto, Ferrofluids: Properties and applications, *Brazilian J. Phys.* **35** (718) 2005, `http://dx.doi.org/10.1590/S0103-97332005000400018`

2. E. N. Velichko, G. L. Klimchitskaya and E. K. Nepomnyashchaya, Casimir repulsion though water-based ferrofluid, *Mod. Phys. Lett. A* **35** (2020) 2040016, `http://dx.doi.org/10.1142/S0217732320400167`

3. E. N. Velichko, G. L. Klimchitskaya and V. M. Mostepanenko, Dispersion forces between metal and dielectric plates separated by a magnetic fluid, *Tech. Phys.* **64** (2019) 1260, `http://dx.doi.org/10.1134/S1063784219090214`

4. G. L. Klimchitskaya, V. M. Mostepanenko, E. K. Nepomnyashchaya and E. N. Velichko, Impact of magnetic nanoparticles on the Casimir pressure in three-layer systems, *Phys. Rev. B* **99** (2019) 045433, `http://dx.doi.org/10.1103/PhysRevB.99.045433`

5. E. A. Perigo, G. Hemery, O. Sandre, D. Ortega, E. Garaio, F. Plazaola and F. J. Teran, Fundamentals and advances in magnetic hyperthermia, *App. Phys. Rev.* **2** (2015) 041302, `http://dx.doi.org/10.1063/1.4935688`

6. E. K. Nepomnyashchaya, E. N. Velichko, I. V. Pleshakov, E. T. Aksenov and E. A. Savchenko, Investigation of ferrofluid nanostructure by laser light scattering: medical applications, *J. Physics: Conference Series* **841** (2017) 012020, `http://dx.doi.org/10.1088/1742-6596/841/1/012020`

7. F. Shi, X. Bai, F. Wang, F. Pang, S. Pu and X. Zeng, All-fiber magnetic field sensor based on hollow optical fiber and magnetic fluid, *IEEE Sensors Journal* **17** (2017) 619, `http://dx.doi.org/10.1109/JSEN.2016.2636879`

8. Kun Ren, Xiaobin Ren, Yumeng He and Qun Han, Magnetic-field sensor with self-reference characteristic based on a magnetic fluid and independent plasmonic dual resonances, *Beilstein J. Nanotechnol.*, **10** (2019) 247, `https://doi.org/10.3762/bjnano.10.23`

9. Q.-F. Dai, H.-D. Deng, W.-R. Zhao, J. Liu, L.-J. Wu, S. Lan, and A. V. Gopa, All-optical switching mediated by magnetic nanoparticles, *Opt. Lett*. **35** (2010) 97, `https://doi.org/10.1364/OL.35.000097`

10. S. Y. Yang, J. J. Chieh, H. E. Horng, C.-Y. Hong and H. C. Yang, Origin and applications of magnetically tunable refractive index of magnetic fluid films, *Appl. Phys. Lett.* **84** (2004) 5204, `https://aip.scitation.org/doi/10.1063/1.1765201`

11. A. Candiani, M. Konstantaki, W. Margulis and S. Pissadakis, A spectrally tunable microstructured optical fibre Bragg grat-

ing utilizing an infiltrated ferrofluid, *Opt. Express* **18** (2010) 24654, https://doi.org/10.1364/OE.18.024654

12. W. Liao, X. Chen, Y. Chen, S. Pu, Y. Xia and Q. Li, Tunable optical fiber filters with magnetic fluids, *Appl. Phys. Lett.* **87** (2005) 151122, https://doi.org/10.1063/1.2103401
13. A. Candiani, W. Margulis, C. Sterner, M. Konstantaki and S. Pissadakis, Phase-shifted Bragg microstructured optical fiber gratings utilizing infiltrated ferrofluids, *Opt. Lett*. **36** (2011) 2548, https://doi.org/10.1364/OL.36.002548
14. H. V. Thakur, S. M. Nalawade, S. Gupta, R. Kitture and S. N. Kale, Photonic crystal fiber injected with Fe3O4 nanofluid for magnetic field detection, *Appl. Phys. Lett*. **99** (2011) 161101, https://doi.org/10.1063/1.3651490
15. P. Zu, C. C. Chan, T. Gong, Y. Jin, W. C. Wong and X. Dong, Magneto-optical fiber sensor based on bandgap effect of photonic crystal fiber infiltrated with magnetic fluid, *Appl. Phys. Lett*. **101** (2012) 241118, https://doi.org/10.1063/1.4772017
16. H. E. Horng, J. J. Chieh, Y. H. Chao, S. Y. Yang, CH.-Y. Hong and C. Yang, Designing optical-fiber modulators by using magnetic fluids, *Opt. Lett*. **30** (2005) 543, https://doi.org/10.1364/OL.30.000543
17. E. K. Nepomnyashchaya, A. V. Prokofiev, E. N. Velichko, I. V. Pleshakov, Yu. I. Kuzmin, Investigation of magneto-optical properties of ferrofluids by laser light scattering techniques, *J. Magn. Magn. Mat.,* **431** (2017) 24, https://doi.org/10.1016/j.jmmm.2016.10.002
18. P. C. Scholten, The origin of magnetic birefringence and dichroism in magnetic fluids, *IEEE Transactions on Magnetics* **16** (1980) 221, https://doi.org/10.1109/TMAG.1980.1060595
19. M. G. Shlyagin, P. M. Agruzov, I. V. Pleshakov, A. V. Prokofiev and E. E. Bibik Incident-power-dependent refractive index of ferrofluid in magnetic field measured with a fiber optic probe, *Optik* **186** (2019) 418, https://doi.org/10.1016/j.ijleo.2019.04.130
20. E. E. Bibik, B. Ya. Matygullin, Yu. L. Raikher and M. I. Shliomis, Magnetostatic properties of magnetite colloids, *Magnetohydrodynamics* **9** (1973) 61, http://doi.org/10.22364/mhd
21. P. M. Agruzov, I. V. Pleshakov, E. E. Bibik and A. V. Shamray Magneto-optic effects in silica core microstructured fibers with a ferrofluidic cladding, *Applied Physics Letters* **104** (2014) 071108, http://doi.org/10.1063/1.4866165
22. P. M. Agruzov, I. V. Pleshakov, E. E. Bibik, S. I. Stepanov and A. V. Shamrai, Transient magneto-optic effects in ferrofluid-filled microstructured fibers in pulsed magnetic field, *Europhysics Letters* **111** (2015) 57003, https://doi.org/10.1209/0295-5075/111/57003