HYBRID FIBER-OPTIC/NANOFIBER DETECTION PRINCIPLE FOR SECURITY APPLICATIONS

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Abstract
In this paper, a novel patent-pending approach based on optical detection will be described. Various functionalized nanofiber materials have been used to demonstrate feasibility of realization of miniature sensors of biomedical and chemical values (enzymes reactions, metal ions content, etc.). Compactness and sensitivity of the sensors are significantly enhanced through original hybrid fiber-optic/nanofiber design. The potential of the new detection principle for security applications (forensic, seal intrusion, etc.) will be discussed.

Keywords: nanofibers, nanotechnology, fiber optics, security, forensic, seal, sensors

1. *Introduction*

Nanofibers are known for their exceptional surface area and wide opportunities for their functionalization. These properties have been attractive for various sensor applications, however, mostly electric sensing principles have been reported.
Fiber optic sensors (FOS) represent most significant non-communication application of optical fibers [1-3]. In comparison to the other sensor types, they excel especially in high sensitivity, geometrical variability and suitability for the use at high voltage and temperature, explosive, corrosive and flammable environment, high electromagnetic interference, etc. Their compatibility with modern fiber optic transmission systems is also a great advantage.
Functional principle of FOS is modulation of optical signal carried by an optical fiber, caused by the detected/measured physical (or another) environmental effect.

2. *Principles of Fiber Optic Sensors*

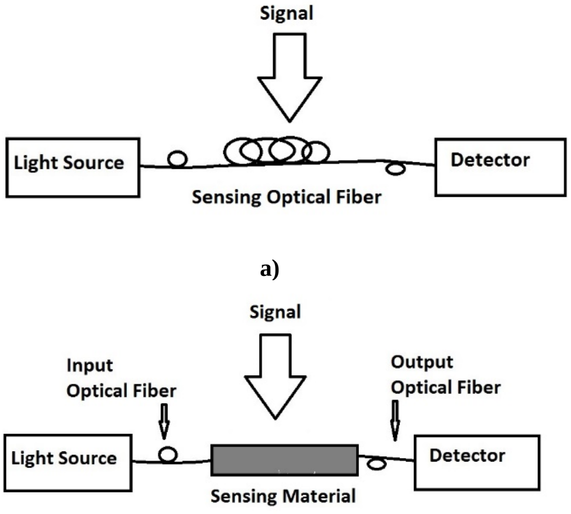
According to the light modulation method, the FOS can be divided into [1-3]:

a) Intensity (amplitude) FOS using influence of the measured value to intensity of the light propagated in the optical fiber,
b) Polarization FOS based on the effect of the measured value to the bi-refrigence of a single-mode optical fiber, resulting in a rotation of polarization plane of the transmitted optical signal,
c) Phase (interferometric) FOS based on phase modulation of the electromagnetic wave propagated in the optical fiber, most often due to photo-elastic effect,
d) Frequency (wavelength) FOS, often using a shift of absorption threshold of suitable detection material (e.g. semiconductor) [1-3, 7, 9],
e) Propagation time of optical pulse in a fiber (methods based on Optical Time Domain Reflectometry – OTDR).

All the above mentioned modulation principles can be realized in the optical fiber itself or outside the fiber. Thus, FOS can be divided into two main groups:

- Intrinsic FOS (optical fiber is the sensing element)
- Extrinsic FOS (optical modulation occurs outside of the optical fiber)

Schemes of the intrinsic and extrinsic FOS are shown in Fig. 1.



a)

b)

Fig. 1 Intrinsic (a) and extrinsic (b) design of fiber optic sensors

Intensity modulation method is most widely used because of its simplicity and sufficient sensitivity.
Several principles of intensity modulation FOS have been reported in both intrinsic and extrinsic categories. Large family of intensity FOS benefits from breaking the total internal reflection condition

$$\theta \geq arc\sin\frac{n_2}{n_1} \qquad (1)$$

where θ is incident angle, n_2, refractive index of fiber core. If the refractive index n_1 of the fiber cladding or incident angle is changed (e.g. due to fiber bending), intensity of transmitted light is influenced. Examples of the use of this principle are illustrated in Figures 2 to 5. FOS shown in Figure 2 is based on temperature dependence of a portion of the cladding of the sensing optical fiber [5]. Simple modulation method using microbending (figure 3) of the sensing optical fiber can be used to design various FOS of mechanical (directly) or other (indirectly) values [6].

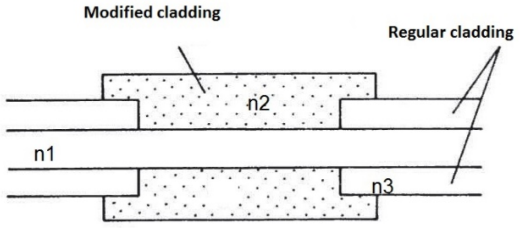


Fig. 2 Fiber optic temperature sensor using modified cladding of sensing optical fiber

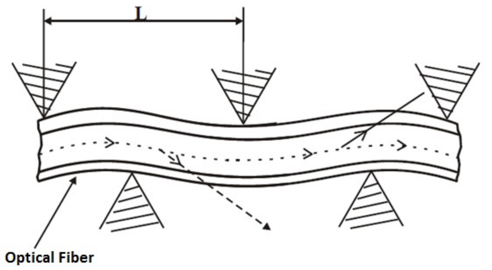


Fig. 3 Microbend intensity modulation in multimode optical fiber

An example of extrinsic FOS based on temperature dependence of light absorption in a semiconductor is shown in figure 4.

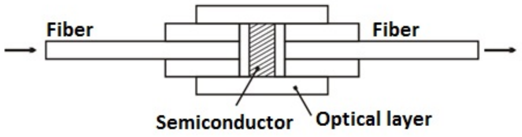


Fig. 4 Extrinsic FOS of temperature

Extensive research effort has been dedicated to development of **polarization FOS** based on Faraday's effect in a single-mode polarization-maintaining fiber. Main motivation was the opportunity to measure electric currents in high voltage power lines. A coil from the optical fiber around the line (figure 5) is an ideal tool for a safe non-galvanic device detecting magnetic component of the electromagnetic field that is proportional to the measured current. Polarization plane of the light propagated in the fiber is turned according to the equation (2), which is easily transformed to intensity change using a polarizer at the fiber output.

$$\theta_F = V\int_0^L \boldsymbol{H}\,d\boldsymbol{l} \tag{2}$$

where θ_F is angle polarization plane is turned due to the magnetic field intensity $\boldsymbol{H}$, L is total length of the optical fiber in the coil and V is Verde constant.

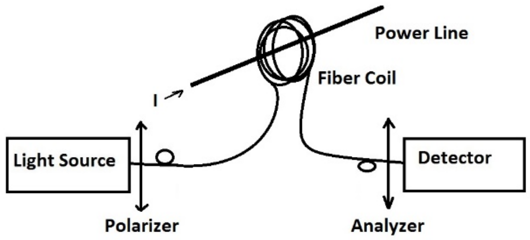


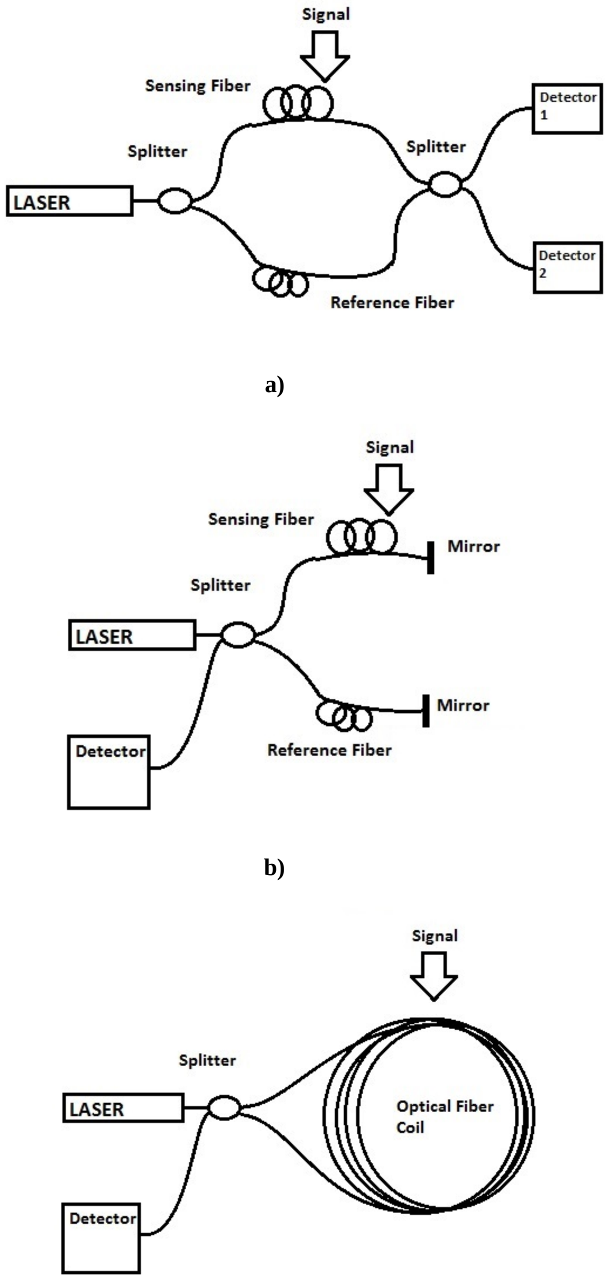
Fig. 5 Polarization intrinsic FOS of electric current

Interferometric FOS are extremely sensitive devices capable to detect phase changes of the order of 10^{-8} rad. This corresponds for example to detection threshold of interferometric fiber optic magnetometers at 10^{-13} T, or temperature changes of 10^{-8} K [1-3, 7, 9]. Phase shift $\Delta\emptyset$ caused by the signal Δx can be generally expressed as

$$\Delta\emptyset = \left(nk\frac{dL}{dx} + kL\frac{dn}{dx}\right)\Delta x \tag{3}$$

n is refractive index, k – wave number and L interaction length of the optical fiber.

Fiber optic interferometers can be very compact and robust in comparison with bulk optics constructions. Three basic types of fiber optic interferometers are illustrated in figure 6.



a)

b)

c)

Fig. 6 Fiber optic interferometers: a) Mach-Zehnder, b) Michelson, c) Sagnac

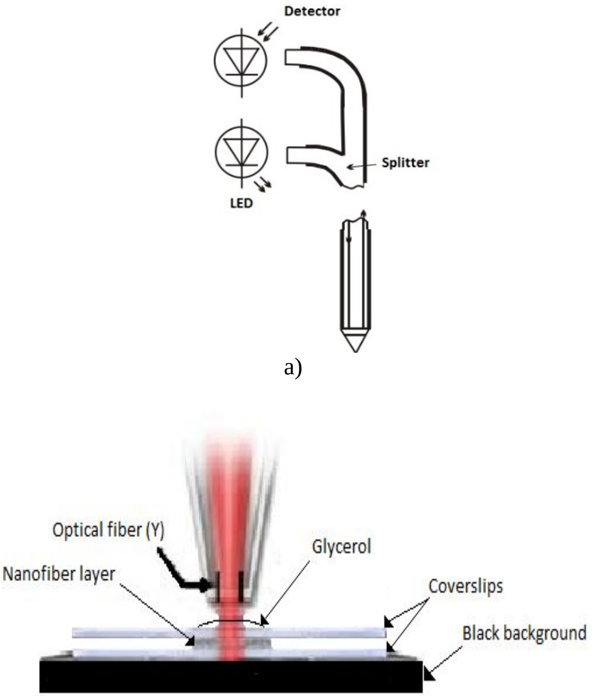
Several types of FOS matured to commercial stage. Recently, some methods (originally developed for service purposes) have been successfully applied as a basis for FOS. For example, Optical Time Domain Reflectometry (OTDR) methods and equipment are being installed as distributed sensor systems for construction (bridges) and oil/gas pipes monitoring [10].

Interdisciplinary research is focused on the use of new materials, including nanomaterials. Nanofibers can be used as evanescent lightguides sensitive to environmental influences [11]. Functionalized nanofibers are being tested as sensing materials for biomedical and chemical FOS [12, 13].

3. *Experimental*

Hybrid fiber-optic/nanofiber detection principle has been verified using the opto-electronic system shown in figure 7. Plastic optical fiber components ("Y" junction with optical connectors) are attached to the proprietary electronic system consisting of red LED as light source and a photodiode as detector. Functionalized nanofiber mat has been attached at the end of the detection fiber (shown in detail in figure 8). Light emitted by the LED is carried to the sensing point and partially reflected back. The portion of the reflected light is measured by the photodetector.

Fig. 7 Opto-Electronic System



a)

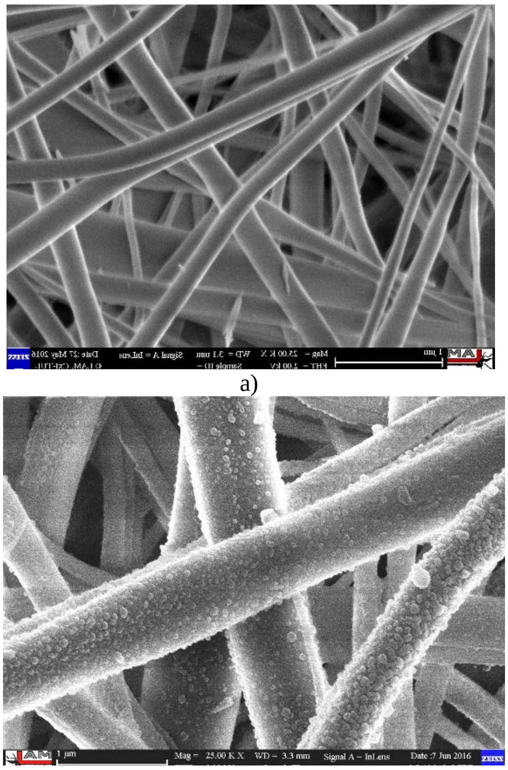
b)

Fig. 8 Detection part of the sensor (a – optical scheme, b – realization)

As a model sensing nanofiber material, we used SiO_2 nanofibers prepared by electrospinning method on the commercial Nanospider™ NS 1S500U machine [14]. The spinning process is illustrated in figure 9.

Fig. 9 Nozzle-less electrospinning process inside Nanospider™ machine

Enzyme Estarase (Esterase from porcine liver (EC. 3.1.1.1.)) and amino acid Thyrosine have been immobilized at the surface of the nanofibers with the help of Glutaraldehyde. Deposition of the enzyme at the nanofiber surface is demonstrated in figure 10.



a)

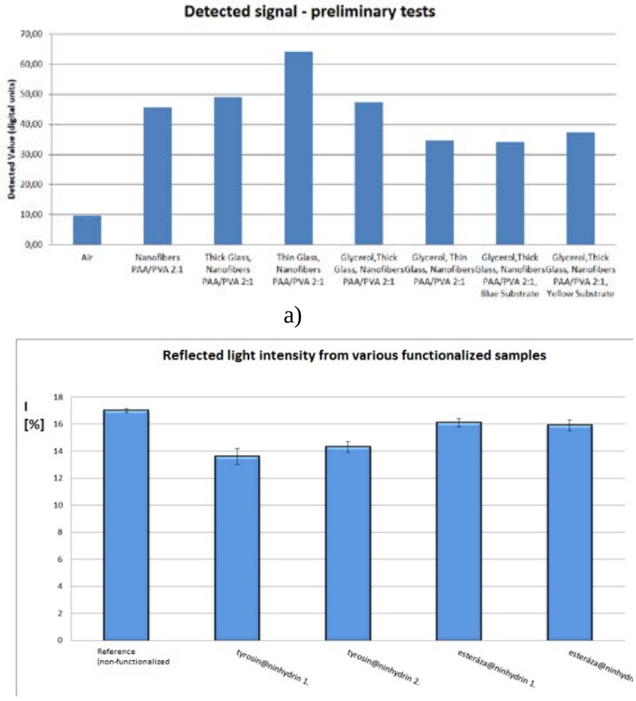
b)

Fig. 10 SEM pictures of nanofibers without (a) and with (b) immobilized enzyme

Subsequently, the samples were immersed into a solution reacting with the enzyme. All reactions were done in Phosphate buffer of pH 7,2. For enzyme-histology proof, substrate of α-Naphtyl acetate (dissolved in 1 ml of acetone) was used; for visualization: O-DIANISIDINE (Sigma-Aldrich) and FAST BLUE RR SALT (sigma-Aldrich) were used. For amino acid proof, reaction with Ninhydrin was used and heat up to 110°C for visualization. Reflected light from the sensing nanofiber material has been recorded.

4. *Results and Discussion*

Initially, several construction parameters (nanofiber materials combinations, background, glass thickness, etc.) were varied to test reproducibility of the measurements and to estimate detection ability of the system. Results of these preliminary tests are shown in figure 11, which demonstrates sufficient response of the system to subtle changes of optical conditions at the tip of the sensing optical fiber. Figure 11b illustrates, that there are measurable differences between optical responses from nanofiber materials functionalized with enzymes and the reference (pure) nanofibers.



a)

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Fig. 11 Reflected light intensity: a - preliminary tests, b - from various fuctionalized nanofiber materials

Detected signal (reflected light intensity) as a function concentration of a model enzyme-substrate is given in figure 12. It is evident that there is a detection limit near 10 % of full initial level.

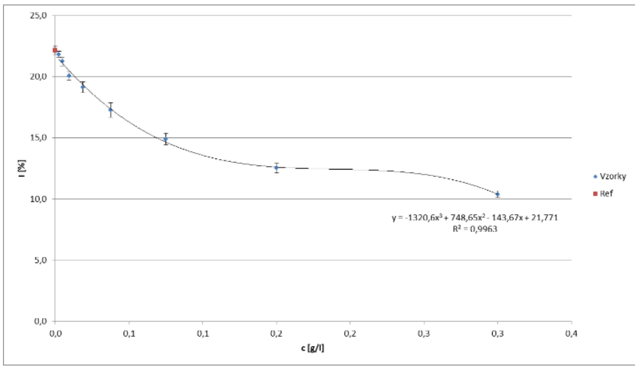

Fig. 12 Reflected intensity vs. concentration of a model enzyme-substrate

5. *Conclusion*

The experiments proved that the described fiber optic system with the nanofiber math as a detection element can be used as a basis of wide family of fiber optic sensors sensitive to various chemical and biological substances. The proposed fiber optic sensors can be very compact and miniature which enables their intergability into security systems for fast and cost effective detection of biological and chemical traces.

Main advantages of the approach are:

- Chemically inert materials – possibility to disinfect/sterilize
- Miniature dimensions
- Sufficiently high sensitivity

Further activities of this research will be focused on

- Optical design optimization
- Electronic system stabilization and sensitivity adjustment

Acknowledgement

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