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Influence of composition of the magnetic composite coating on the performance of the optical fiber magnetic field sensing element

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The optical fiber magnetic sensing element (OFMSE) based on the optical fiber coated with composite coating polymer–magnetic powder has been already introduced in magnetic field sensing. In this research, the rapid quenched Nd-Fe-B powder with Nd rich content was used as magneto-active component in composite coating. Magnetic powder was dispersed in poly-(ethylene-co-vinyl-acetate) – EVA solution in toluene. The influence of magnetic powder content (20, 30, 40 and 50 mass %) in composite coating of multimode optical fiber on the thickness, hardness and uniformity was observed and discussed. Sensitivity and reversibility of the constructed OFMSE in the external magnetic field are illustrated.

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1. Introduction

The optical fibers provide a large band width, low cost in mass production and low transmission loss in communication channels [1-3]. The standard optical fiber has a shape of long, cylindrical wave guide with different types of core/cladding size and shape. Modification of optical fibers by application of different coatings can alter their propagation characteristics and general behavior of the material in different environments. Depending on the future use lot of composite materials can be applied as a composite coating. The composite coating with the magnetic material as an active component might be used as a sensing element for detection of external magnetic field. Over the years the high sensitivity magnetic field measurements have been utilized to detect the presence of large ferromagnetic objects which change the magnetic field distribution. The optical fiber coated with composite polymer-magnetic powder coating can be used as an optical fiber magnetic sensing element (OFMSE). The measurements using optical fiber are described as the light propagates in the cylindrical optical wave guide with core and cladding modes, the external excitation disturbs the modes to change their modes to change their propagation properties which can be detected and measured. The advantage of the OFMSE is that it can be used in areas that are too harsh for measurements with a conventional sensor system, since the optical fibers are usually made of dielectric material that have high resistance to vibration, electromagnetic interference, thermal shock and corrosion. [4-5].

The OFMSE in this study is intensity modulated displacement optical fiber sensing element. The principle components of the OFMSE are a pair of spaced apart optical fibers and at least one of which forms a cantilevered beam and has a composite coating. Fluctuations of the magnetic field, which is orthogonal to the longitudinal axis of the cantilevered optic fibers, will cause fluctuating mechanical response in magnetic composite coating, which will be transferred to the cantilevered optical fiber. Deflection of the coated cantilevered beam optical fiber will produce fluctuation coupled light to the other optical fiber with such fluctuations being proportional to the applied magnetic field. It is obvious that the properties of the composite coating will have the great influence of performance of the OFMSE.

A quasi-distributed, optical-fiber sensing (QDOFS) system is one for which only prescribed section of the fiber is sensitive to the measuring field. These systems have advantages that only those prescribed sections have any sensitivity to the measured field and that their positions are known, the disadvantage is that the required measurement points in the field must be known in advance [6]. The OFMSE investigated in this study to a certain degree belongs to this type of sensing system, because only the end of one optical fiber is coated and therefore sensible to the magnetic field.

The composite coating can be made by adapting the existing process of manufacturing optical fibers in stage in which polymer coating is applied to the drawn fiber. Instead of solely polymer coating, coating with particles of magnetic powder can be used. After serial of experiments of investigation of the influence of coating velocity and concentration of magnetic powder in polymer on uniformity of coating, the processing parameters for coating of optical fiber with magnetic coating were defined [7-9]. Appropriate composite coatings should be homogenous and thus enable reliable magnetic detection, while minimizing the side effects. A copolymer of ethylene and vinyl-acetate (EVA) was chosen for the polymer component of the composite coating, because of its good adhesive properties. By using EVA, it is possible to produce coating without the application of UV or thermal curing process, and hence the number of process parameters was reduced. The magnetic component of the composite coating can be selected from a variety of permanent magnetic powders (hard ferrite, Sm-Co, Nd-Fe-B) [10-12]. Magnetic properties and behavior of this type of magnetic alloys were observed in previous investigations [13-16].

For this investigation, magnetic powder of Nd –rich Nd-Fe-B alloy was chosen.

In previous investigations, it was experimentally determined that micromechanical and propagation properties of constructed sensing element depend upon the type, characteristics and the amount of magnetic powder in the composite coating and on quality of coating itself (uniformity, thickness and micromechanical properties) [5]. The influence of the amount of the applied magnetic powder in the composite coating of multimode optical fiber on the thickness, hardness and uniformity was observed and discussed in this work. Propagation characteristics of the constructed OFMSE in the external magnetic field in dependence of quality and composition of applied composite coatings are illustrated also.

2. Experimental

2.1 Characterization of the powder

As a magnetic component of composite coating, magnetic powder of Nd rich Nd-Fe-B (32 mass %) alloy synthesized by method of rapid quenching from mold was used. Dimension mean value of starting Nd-Fe-B powder particles was between 100 and 150 μm. In order to use it as a magnetic component in composite coating of optical fiber for constructing sensing element, Nd-Fe-B magnetic powder was milled to the appropriate particle size. In previous research, it was observed that particle size of magnetic powder has big influence to the homogenousity of suspension polymer – magnetic medium, and then to the uniformity of composite coating on the optical fiber. During research, it was found that optimal size of magnetic particles is between 1 and 10 μm. Morphological tests were done on the optical microscope “Reichart”, type POLYVAR – MET with reflected light. Analysis of picture was done on the automatic equipment Q500MC – Leica with appropriate software [5]. Nd-Fe-B powder was milled under protecting, inert fluid toluene for 2.5 hours. After finished milling, powder was dried at room temperature in inert atmosphere of argon. SEM photo of Nd-Fe-B powder before and after milling is presented on Fig. 1.

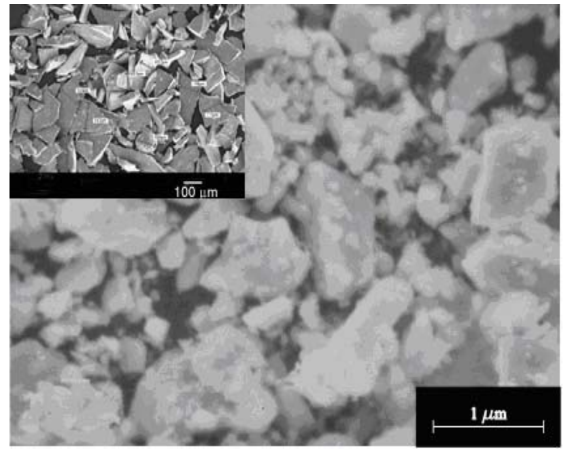


Fig. 1. SEM photo of rapid quenched Nd-Fe-B magnetic powder before (insert figure) and after milling.

Metallographic measurements and morphological analysis were done by measuring size of the particles, their percent ratio and distribution. Each morphological measurement was done on 600–700 particles. Due to the fact that particles are magnetic which caused formation of agglomerates, it was harder to do morphological testing on more particles in the visible field of microscope. In order to observe shape of the particles, maximal, minimal diameter (D_{max}, D_{min}) was measured as well as surface (A) and perimeter (L_p) of particles. Total statistical data were presented in Table 1.

Table 1. The statistical data for measurements of Nd-Fe-B particles dimensions after 2.5 hours of milling in toluene.

	Mean value	Min. value	Max. value	RSE (%)
A (μm^2)	0.52959	0.1335	1.2425	3.25899
L_p (μm)	2.93917	1.5523	5.625	2.30133
D_{max} (μm)	0.9148	0.4902	1.54695	2.11377
D_{min} (μm)	0.68922	0.1634	1.4756	2.10238
Ff	0.65605	0.129	0.895	0.96639

Results of morphological analysis show that approximatelly 50 % of particles have diameter about 1 μm and that sizes of 90% of particles are below 5 μm. Metallographic analysis (Fig. 1) and obtained values of perimeter form factor (Ff) after the milling suggest that the roundness of particle shape is increased and that the particles have the shape closer to ellipse or circle that to dendrite. The Nd-Fe-B powder prepared via this route was used as the magneto-active component in the process of preparation of composite coating.

2.2 Coating of optical fiber

The original coating of optical fiber was removed and polymer – magnetic composite coating was applied instead of it. The applied polymer – magnetic composite coating is the mixture of permanent magnetic powder and EVA - poly (ethylene-co-vinyl acetate) produced by DuPont under commercial name ELVAX 265. Polymer was used in a form of a toluene solution with 33.33 mass% of polymer. After dissolving EVA in toluene, magnetic powder was dispersed in the solution. Procedure for construction of the OFMSE was:

- Milling of magnetic powder in toluene
- Dissolving of ELVAX 265 at 60 ºC during 2.5 hours
- Dispersion of magnetic powder in EVA solution
- Homogenization by mechanical and ultrasound mixing
- Removing of original coating of optical fiber by flaming
- Applying of composite coating on the bare optical fiber
- Removing of the drop that is formed at the end of fiber
- Construction of the OFMSE

Samples with four different composite coating compositions were made with mass fraction of magnetic powder: 50, 40, 30 and 20 % corresponding to volume fraction of 11.43, 7.92, 5.24 and 3.12% respectively.

2.3 Microhardness measurements

The composite coating and optical fiber can be considered as hybrid composite, and the hardness can be investigated in regard of relative indentation depth (RID) [17]. This is a dimensionless parameter β, given by ratio between the indentation depth, δ, and the coating thickness, t. Values of $\beta > 1$ signify that the indenter severely deformed and penetrated the coating, and reached the underlying substrate material- glass optical fiber. The system response is then dominated by substrate properties, and mainly invariant with further load increase. At the other extreme, experiments show that when $\beta < 0.1$, the influence of the substrate on the deformation is small, and for this and lower indentation depths the coating-only response is observed [18].

For a given hardness value, a one-to-one relationship exists between the load P and maximum indentation depth δ given by:

$$H = \frac{\kappa \cdot P}{\delta^2} \tag{1}$$

where κ is a parameter describing the indenter geometry. For micro-Vickers indenter geometry:

$$\delta = \frac{d}{7} \tag{2}$$

where *d* is indentation diagonal.

Vickers - indentation microhardness measurement is a well known and reliable test method for the evaluation of mechanical characteristics of coatings. The mechanical properties of the composite coating were characterized using Vicker's microhardness tester ”Leitz, Kleinharteprufer DURIMET I” using loads of 0.4905N and 0.981N. Three indentations were made at each load, yielding six indentation diagonals measurements, from which the average hardness could be calculated. Indentation was done at room temperature.

2.4 Investigation of the sensitivity of coated optical fiber in the external magnetic field

In order to avoid influence of positioning and connecting to the sensitivity of the OFMSE in the capillary tube, plate for simulation has been constructed [11]. Simulation plate consisted of one plastic plate with incision and round hole in the middle. The coated optical fiber and the optical fiber –receiver of signal were positioned in the incision and held in close proximity to each other. Core diameter of coated optical fiber was 62.5µm and the outer diameter was 250µm. The receiving fiber had the core diameter of 200µm and the outer diameter of 400µm in order to ensure better reception of signal during the investigation of magnetic field sensing element.

The light from light emitting diode (λ=849 nm) is launched to an optical fiber with deposited composite coating. Intensity and wavelength of light emitted trough optical fiber were constant during experiment. The light signal was received by the optical fiber that was fixed at the other end of the plate. A photo detector detected the intensity of the light from this fiber. The amplified output signal from this fiber is connected to the data acquisition system based on the A/D card, personal computer and specially developed software in Pascal.

If the OFMSE is not in the magnetic field, the ends of optical fibers are collinear and the intensity of the propagated light is maximal. If permanent magnet is approached to the sensing element, the light intensity decreases.

The propagation properties of the OFMSE with different composition of coating were investigated and the composite coated fiber with the best perfomances was used for construction of the OFMSE in glass capillary tube. After that the sensing element was investigated in presence of external magnetic field.

3. Results and discussion

Measuring of composite coating thickness and uniformity on the optical fiber was done on the microphotographs, using ImagePro picture analysis program. The results of coating thickness determination are presented on Table 2.

Table 2. The results of coating thickness measurements.

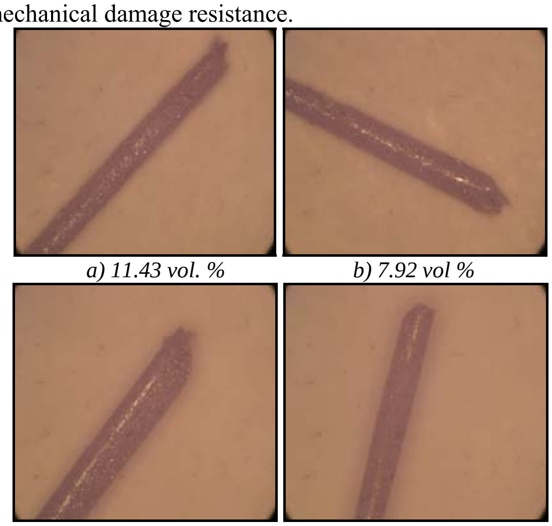
Sample	Fraction of Nd-Fe-B powder (vol %)	The most frequent value of thickness of coating (μm)
1	11.43	45
2	7.92	55
3	5.24	70
4	3.12	70

As ELVAX shows highly viscoelastic properties it was only possible to measure the hardness of coatings with high amounts of magnetic powder. The measurements were done on the optical fiber with investigated composite coating. Thus, this configuration might be observed as a hybrid composite and the results of microhardness measurements in regard to RID values were presented in Table 3:

Table 3. The results of microhardness measurements.

Sample	RID (β)	Hardness (GPa)
1	0.062	4.78
2	0.053	3.76
3	0.099	1.78

The values of $\beta < 0.1$ signify that the measured microhardness is microhardness of coating. As thickness depends on the amount of magnetic powder in composite coating, and microhardness depends on a thickness of composite coating, it may be concluded that microhardness depends on the amount of magnetic powder in the composite coating. The coating with the highest content of magnetic powder shows the highest microhardness and the yielding point, consequently. So, the coating with this composition possesses the highest mechanical damage resistance.



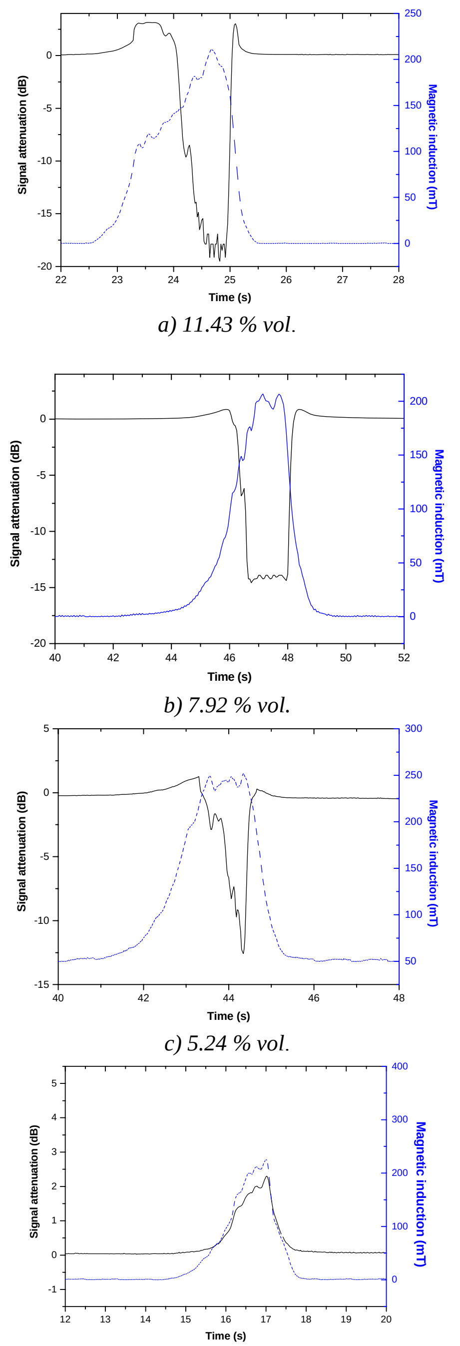
a) 11.43 vol. % *b) 7.92 vol %*

c) 5.24 vol % *d) 3.14 vol %*

Fig. 2. Microphotographs of optical fibers with composite coating with different amounts of magnetic powder in composite coating, × 50.

Microphotographs of coated optical fiber ends used for construction of the OFMSE with different composite coating composition are presented on Fig. 2.

The results of measurements of signal attenuation are presented on Fig. 3. It should be noted that viscoelastic behavior of hybrid composite is sensitive to the amount of magnetic powder in composite coating. Delay of the OFMSE response to the magnetic field increases with decreasing of magnetic powder content in composite coating.



a) 11.43 % vol.

b) 7.92 % vol.

c) 5.24 % vol.

d) 3.12 % vol.

Fig. 3. The response of the samples with different amount of magnetic powder in external magnetic field.

The optical fiber magnetic sensor in this study is based on the principle of intensity modulation and consists of two optical fibers held in close proximity to each other.

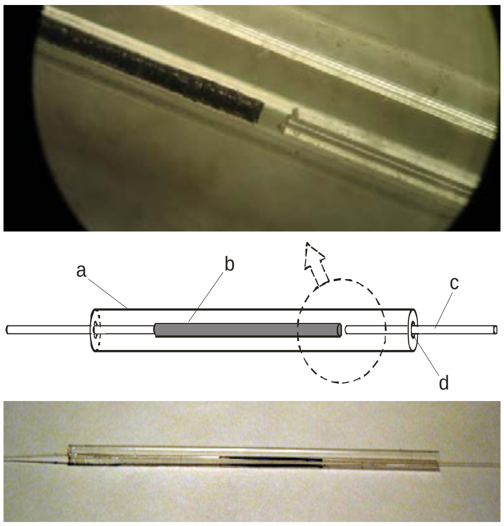


Fig. 4. Scheme of the OFMSE (a – capillary tube, b – fiber coated with polymer magnetic coating, c – optical fiber, d – glue) and photos of constructed OFMSE.

One of those optical fibers (one coated with magnetic composite) is cantilevered in a glass capillary tube. The cantilevered section moves in the opposite direction to the rest of sensor in response to an applied external magnetic field, and consequently the amount of light coupled between two fibers is modulated [6]. Scheme of the OFMSE is presented on Fig. 4.

Based on previously mentioned results [8,11], sensing element in capillary tube was constructed, using optical fiber with composite coating with 11.43 % volume fraction of Nd-Fe-B magnetic powder. Design of this element was the same in dimensions with the element that was simulated on the plate, but in this case both optical fibers had the same diameter. Thus the construction and positioning of the optical fibers in the OFMSE was more difficult. The result of testing of this sensing element to the external magnetic field (the strength of output signal of the OFMSE in the presence of the external magnetic filed) is presented on Fig. 5.

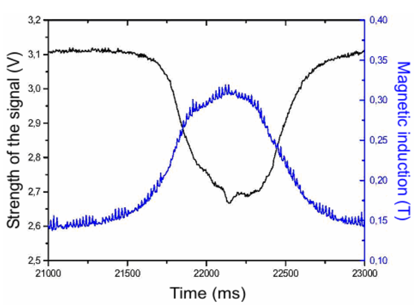


Fig. 5. The response of the constructed OFMSE (11.43 vol % of magnetic powder in composite coating) to the external magnetic field.

The constructed OFMSE has shown good response to the external magnetic field and good reversibility of strength of a signal despite the difficulties that occurred during the assembly concerning the supporting, positioning and colinearity of fibers.

4. Conclusion

The influence of the amount of magnetic powder of Nd-Fe-B type in composite coating on mechanical properties (thickness, uniformity, and hardness) was observed. Increased amount of Nd-Fe-B powder causes the decrease in the thickness of composite coating, due to magnetic cohesion. Comparison of thickness and uniformity of composite coating with different amounts of Nd-Fe-B powder and measured values of hardness, it might be concluded that hardness of composite magnetic coating directly depends on the amount of magnetic medium. The best results in regard to micromechanical and propagation characteristics are gained with the coating with 11.43 vol % of magnetic powder. The sensing element based on modified optical fiber with 11.43 vol. % of Nd-Fe-B magnetic powder was constructed and its propagation characteristics in external magnetic field were investigated. The constructed OFMSE (in capillary tube) exhibites good response to the external magnetic field and good reversibility of strength of a signal. The goal of the future investigation should be in the direction of improvement of sensitivity of OFMSE, possibly by modification of the construction and the arrangement of the optical fibers in the sensing element.

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