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Investigation of the Conditions of Synthesis of Optical Fiber-Magnetic Powder Composites*

A. Milutinović-Nikolić¹, N. Talijan¹, R. Aleksić²

¹Institute of Chemistry, Technology and Metallurgy, Njegoševa 12,
11000 Belgrade, Yugoslavia

²Faculty of Technology and Metallurgy, Karnegijeva 4,
11000 Belgrade, Yugoslavia

Abstract: The goal of these investigations was synthesis of optical fiber-magnetic powder composites with new application possibilities like sensors, detectors etc. Parts of results of the investigation of the possibility of embedding magnetic powder of SmCo_5 type in the coating of optical fiber are presented. The influence of the dispersion composition on the uniformity of composite coating was analyzed. For each investigated concentration of magnetic powder SmCo_5 a composition of dispersion that produced the most uniform composite coating, for constant velocity and temperature of coating was established.

Keywords: Composite; Optical Fiber; SmCo_5 Powder; Coating Conditions.

Резюме: Настоящие исследования направлены на получение композитов оптическое волокно-магнитный порошок с новыми возможностями применения — датчики, детекторы и др. Показана часть результатов возможностей включения в оболочку оптического волокна магнитного порошка типа SmCo_5 . Проанализировано воздействие состава дисперсии на равномерность композитного покрытия. Для каждой исследованной концентрации магнитного порошка SmCo_5 установлен состав дисперсии, способствующей возможности создания самого равномерного покрытия при постоянной температуре и скорости нанесения покрытия.

Ключевые слова: Композит; оптическое волокно; SmCo_5 порошок; условия нанесения покрытия.

Садржај: Ова истраживања усмерена су у циљу добијања композита оптичко влакно-магнетни прах са новим могућностима примене - сензори, детектори и др. Приказан је део резултата испитивања могућности уградње магнетног праха типа SmCo_5 у омотач оптичког влакна. Анализиран је утицај састава дисперзије на униформност композитне превлаке. За сваку испитану концентрацију магнетног праха SmCo_5 утврђен је састав дисперзије која омогућава формирање најравномерније превлаке при константној температури и брзини превлачења.

Кључне речи: Композит; оптичко влакно; SmCo_5 прах; услови превлачења.

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1. Introduction

In a preliminary study the optical fiber - polymer binding - permanent SmCo_5 magnetic powder composite material has been obtained. This material widens the rare-earth permanent magnet applications field [1-2]. It is possible to use these composites, as optical sensors of magnetic fields [3-4], for magnetically detectable telecommunication optical fibers [5], etc.

Nowadays highly reliable optical networks are widely used. Optical networks use optical cables for transmitting information on large distances. Often cables need to be buried or placed under the sea [6]. Precise locating and distinguishing different cables is complex, due to their dielectric nature. Embedding a magnetic marker in a cable and/or fiber is one of the possibilities for improving the reliability of networks [5]. A standard magnet detector can easily detect optical cables with magnetic markers. A magnetic marker can be imposed in an optical fiber by dispersing it in the composite coating.

Covering an optical fiber with a coating that includes a permanent magnetic material is the way to obtain the desired composite. The appropriate composite coating should be homogenous and thus gives reliable magnetic detection, while the side effects are minimized. The aim of more complex investigations [7-9] was to determine the optimal conditions of forming uniform composite coating. Numerous process parameters affect the coating uniformity and circularity: temperature, type and concentration of polymer, type, concentration, size, distribution and orientation of particles of magnetic powder, viscosity of dispersion of magnetic powder in a polymer solution or melt, geometry of orifice, coating velocity etc. [7, 10]. Only parts of these investigations are presented in this paper.

The goal of this research was to define process parameters that enable the formation of a coating of uniform thickness on a single-mode optical fiber.

2. Experimental

The optical fiber-magnetic powder composite was obtained using the following starting materials [8-9]:

- Commercial single-mode optical fiber, produced by Alcatel. Attenuation given by the producer was 0.34 dB/km at $\lambda = 1310$ nm and 0.21 dB/km at $\lambda = 1550$ nm.
- Polymer: poly(ethylene-co-vinyl acetate) – EVA with 28 mass % of vinyl acetate, produced by DuPont, under the commercial name - ELVAX 265.
- Fine magnetic SmCo_5 powder.

The commercial SmCo_5 powder used in this experiment, was prepared by the Reduction /Diffusion process. The chemical composition in mass percent was: Sm = $34.7 \pm 0.3\%$ and Co = $64.8 \pm 0.3\%$ [1]. All powder particles were less than 300 μm in diameter. Only a powder consisting of single crystal particles (approx. 1-10 μm) allows a full expression of magnetic properties – remanence and coercivity [11-14]. It is very difficult to align particles larger than 10 μm , and particles smaller than 1 μm are easily oxidized [15-16]. In the ideal case the particle size is the size of a single crystal without grain boundaries within a particle, with one magnetization axis. To obtain single domain particles milling is a necessary step [11]. The powder was

milled in protective anhydrous toluene and all experiments were carried out with a fine powder of single domain particles between 1.0-12 μm with an average size of 8.1 μm [1, 11].

Fig. 1 shows a flow chart of the experimental procedure. A schematic presentation of the structure of a single-mode optical fiber and optical fiber-magnetic powder composite is given in Fig. 2.

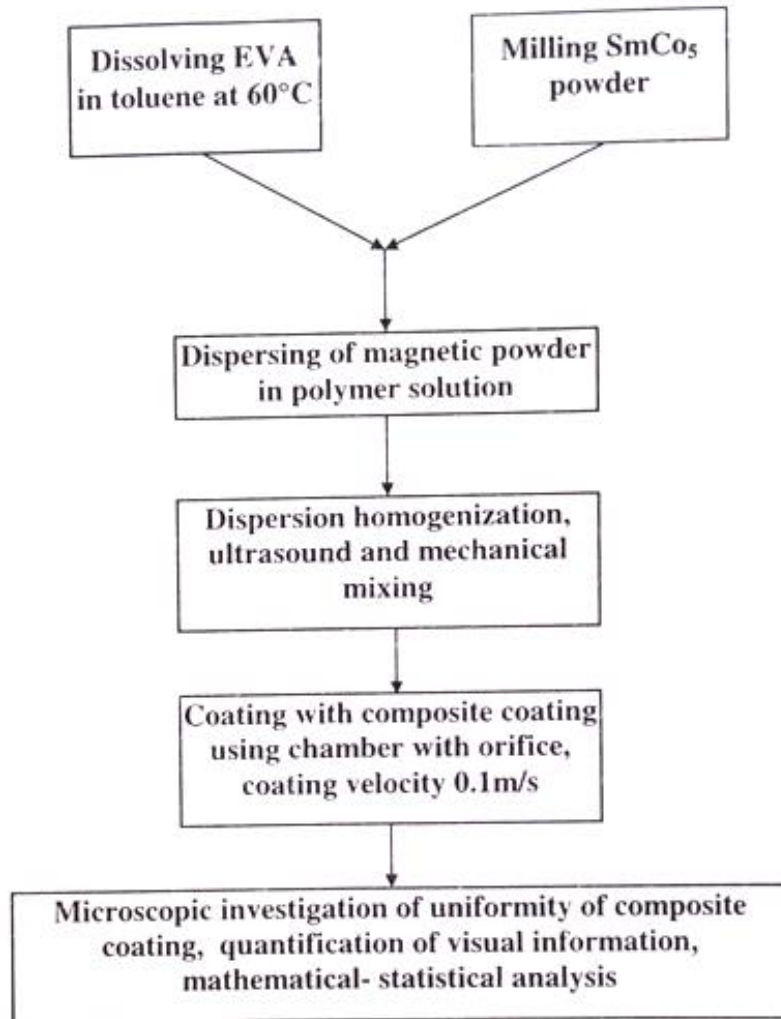


Fig. 1 Flow chart of synthesis of optical fiber-magnetic powder composite

Optical fibers were coated with various dispersions of SmCo₅ in a toluene solution of EVA. First the polymer was dissolved in toluene at 60°C using a magnetic mixer with a heater. Complete dissolving is reached after 2.5 hours. Due to the tendency of gel forming dissolving EVA in toluene at temperatures lower than 60°C is very difficult. At higher temperatures the solution is unstable and splits into solvent-rich and solvent-poorer phases. Therefore 60°C is the optimal temperature for the dissolving process.

After complete dissolving the solution was cooled to working temperature, when the appropriate amount of SmCo₅ powder was added. Ultrasound mixing (5 min) followed by mechanical mixing (30 min) were used to homogenize the

previously obtained dispersion. The composite coating was obtained by pulling the optical fiber through a reservoir with dispersion and an orifice ($d=1.7$ mm) on its bottom. The temperature in the reservoir was controlled and constant ($38\pm 1^\circ\text{C}$). The coating velocity was 0.1 m/s.

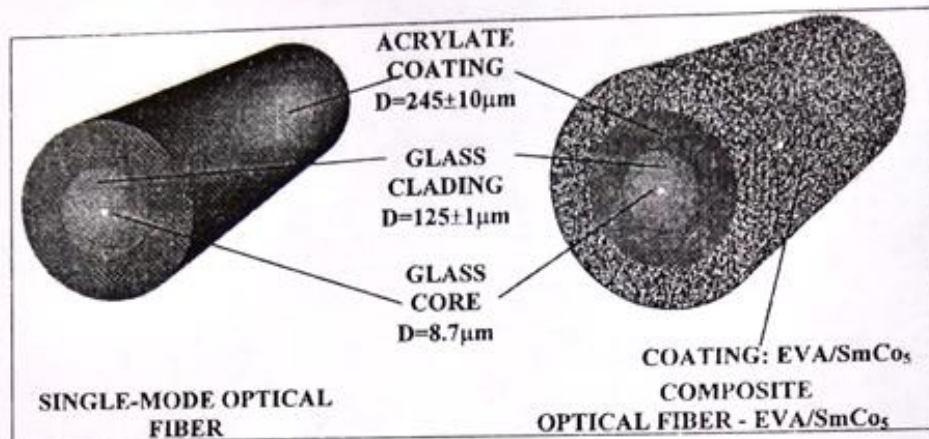


Fig. 2 A schematic structure of a single-mode optical fiber and optical fiber-EVA/SmCo₅ composite

Investigations were performed for a defined content of the magnetic component in the formed composite coating. In a series of pre-experiments it was confirmed that the amount of residual toluene, in the formed composite coating after 24 hours is negligible. Therefore the composition of the composite coating contains solely SmCo₅ and EVA.

The investigation was carried out for a SmCo₅ content of composite coating in the interval from 30 to 60 mass percent. For each investigated concentration of SmCo₅ a composition of dispersion that produced the most uniform composite coating for constant velocity and temperature of coating was established.

A microscope technique was used for measuring the thickness of composite coating. The investigation was carried out on a JENA Carl Zeiss microscope with reflected light. The microscope was equipped with a digital camera connected to a personal computer. The obtained microphotographs were treated with appropriate software for quantification of visual information. The experimental results were evaluated by a mathematical-statistical method.

3. Results and discussion

Uniform coating can be obtained when covering is performed with dispersion of an appropriate viscosity. A special problem arises due to the complexity of dispersion of magnetic powder in the toluene solution of EVA. The viscosity of this system depends on a great number of parameters. The solution of EVA in toluene is a Non-Newtonian fluid with viscosity depending on shear rate and slightly on time besides temperature and polymer concentration. Additional problems arise due to the system tendency of gel forming at lower temperatures. Dispersing magnetic powder in a polymer solution also affects system viscosity. With the increase of magnetic

powder concentration in dispersion viscosity increases. Therefore viscosity of dispersion varies with EVA concentration in toluene, concentration of SmCo_5 powder, temperature, coating velocity, aging, etc.

A series of experiments were performed to obtain the dispersion of the viscosity that provides uniform coating. If the viscosity is smaller than optimal a shape similar to pearls is formed. If the viscosity is greater than optimal inhomogeneous and too thick coatings are formed.

For each investigated concentration of SmCo_5 in the formed coating the dispersion composition that produces the most uniform coating, for a coating velocity of 0.1 m/s and coating temperature of $38 \pm 1^\circ\text{C}$ are presented in Table I, as well as the investigated interval of the dispersion composition.

Table I The influence of dispersion composition on coating uniformity

No.	Investigated interval of dispersion composition			Dispersion composition producing the most uniform coating			Content of SmCo_5 powder in formed coating	
	EVA Mass %	SmCo_5 Mass %	Toluene Mass %	EVA Mass %	SmCo_5 Mass %	Toluene Mass %	Mass %	Vol %
1.	15.0 – 17.0	6.4 – 7.3	75.5–78.5	16.1	6.9	77.0	30.0	4.59
2.	14.5 – 16.5	9.5 – 11.0	72.5–76.0	15.6	10.4	74.0	40.0	6.97
3.	14.0 – 16.0	14.0 – 16.0	68.0–72.0	15.0	15.0	70.0	50.0	10.10
4.	13.0 – 15.0	19.5 – 22.5	63.5–67.5	13.8	20.7	65.5	60.0	14.42

The viscosity of the dispersion composition that produces the most uniform coating thickness in experimental conditions (38°C , coating velocity 0.1 m/s) is in the range 0.7–0.8 Pas.

As an illustration, microphotographs of characteristic coating appearances are presented, as well as the original optical fiber without composite coating (Fig. 3). The photographs were obtained by merging several consequent microscopic images.

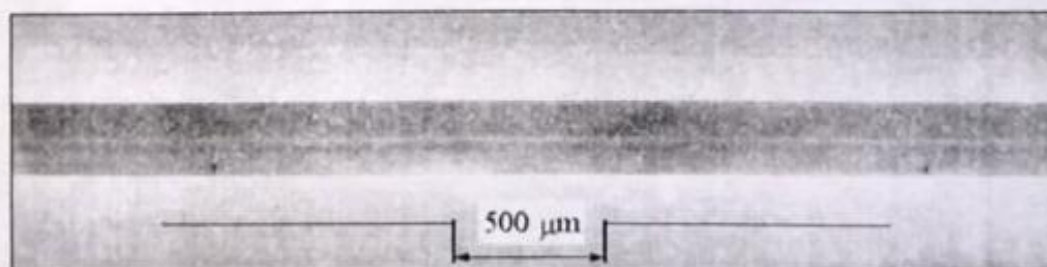


Fig. 3 Microphotograph of optical fiber without composite coating

Figs. 4–6 show microphotographs of optical fibers with characteristic composite coatings depending on coating conditions. The pearl-like coating is shown in Fig. 4, while the coatings with uniform thickness are given in Figs. 5 and 6. The presented content of SmCo_5 in formed coating was 30 mass %, while the composition

of dispersion varied (Figs. 4, 5). The uniform thickness of the coating with 50 mass % of SmCo_5 is presented in Fig. 6.

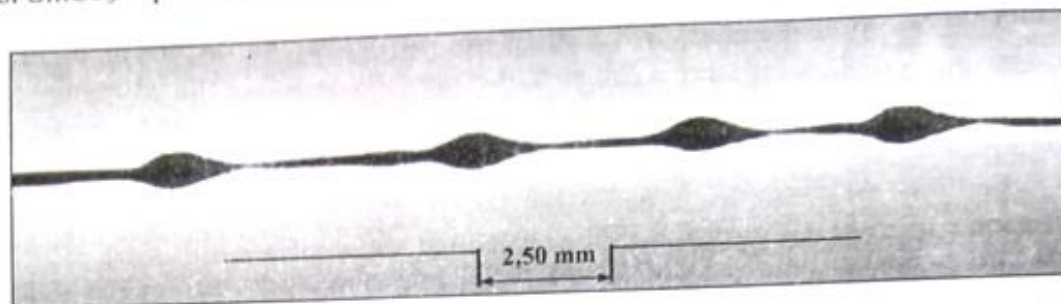


Fig. 4 Photograph of optical fiber with a pearl-like composite coating containing 30 mass% of SmCo_5

The distribution of SmCo_5 powder varies along the fiber (Fig. 5). With the increase of the magnetic component 50 mass % (Fig. 6) the uniformity of distribution of SmCo_5 particles improves. With further increase of the magnetic component the homogeneity within experimental conditions remains approximately the same.

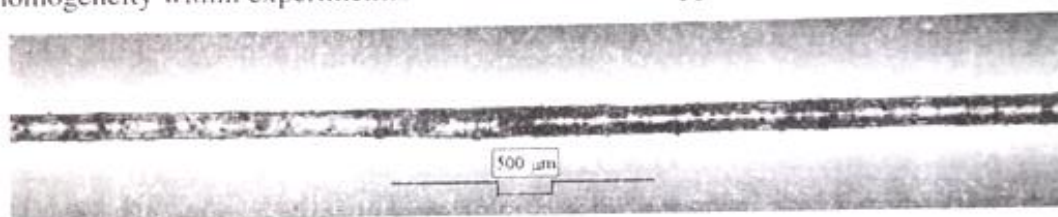


Fig. 5 Microphotograph of optical fiber with uniform composite coating containing 30 mass% of SmCo_5

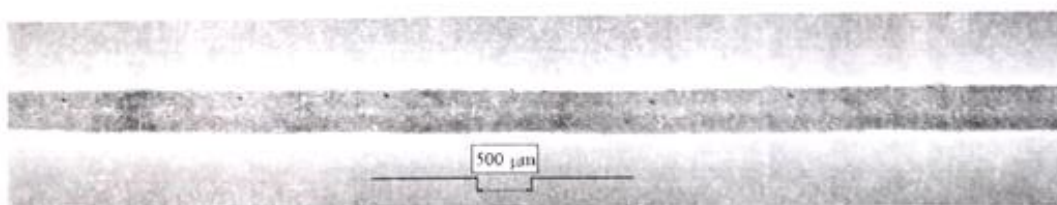


Fig. 6 Microphotograph of optical fiber with uniform composite coating containing 50 mass% of SmCo_5

4. Conclusion

The possibility of embedding magnetic powders in optical fiber coating was analyzed from experimental and theoretical points of view.

A method for making a single-mode optical fiber - magnetic SmCo_5 powder composite (bind with EVA) was developed. Coating optical fiber with a dispersion of SmCo_5 powder in EVA toluene solution was investigated.

The viscosity of dispersion varies with EVA concentration in toluene, concentration of SmCo_5 powder, temperature, coating velocity, aging, etc. Uniform

coating can be achieved if the covering is carried out with dispersion of appropriate viscosity.

For each investigated concentration of SmCo_5 a composition of dispersion that produced the most uniform coating thickness for a constant velocity and temperature of coating is established.

The most uniform distribution of SmCo_5 particles along the fiber were obtained for 50 mass % of SmCo_5 in the formed coating.

Previous experiments [9] suggest that SmCo_5 is a good candidate for a magnetic component of a optical fiber-magnetic material composite with preserved transmission characteristics, useful for magnetically detectable telecommunication optical fibers. Investigations are still in progress. After interferometric, DFB and magnetic measurements a final conclusion about properties of the new material will be presented.

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