# Active Optical Fibers Doped with Ceramic Nanocrystals

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***Abstract.*** *Erbium-doped active optical fiber was successfully prepared by incorporation of ceramic nanocrystals inside a core of optical fiber. Modified chemical vapor deposition was combined with solution-doping approach to preparing preform. Instead of inorganic salts erbium-doped yttrium-aluminium garnet nanocrystals were used in the solution-doping process. Prepared preform was drawn into single-mode optical fiber with a numerical aperture 0.167. Optical and luminescence properties of the fiber were analyzed. Lasing ability of prepared fiber was proofed in a fiber-ring set-up. Optimal laser properties were achieved for a fiber length of 20 m. The slope efficiency of the fiber-laser was about 15 %. Presented method can be simply extended to the deposition of other ceramic nanomaterials.*

## Keywords

***Erbium, nanocrystals, special optical fiber.***

## 1. Introduction

During last fifty years, which have passed from the realization of the first fiber laser [1], fiber lasers have been established as an inherent part of number of advanced photonic devices. Recently, the telecommunication boom; including a rapid expansion of internet; has created challenging request to the data transfer and processing. Since the most of the data transfers are realized on optical fibers, fiber laser have found a crucial place in them; namely erbium doped fiber amplifiers have been established as key components of optical networks [2]. Beside the telecommunications fiber lasers can be found as sophisticated components of high-tech devices that are widely used in data processing systems, frequency and time metrology, mechanical engineering and industrial processes [3]. Nowadays, they have expanded into biology and medicine as powerful tools for researchers, diagnostics and therapy [4].

The design of fiber lasers widely exploit luminescence properties of rare earth elements (REE) entrapped inside core of the optical fiber. REE or their combination primarily determines the operating wavelength of the fiber laser. As an example, a combination of erbium and ytterbium ions is used in lasers for telecommunication at 1.55 μm [5]. High-power lasers usually employ luminescence properties of neodymium or ytterbium ions around 1.05 μm [6].

Various methods and processes have been developed to produce fiber suitable for fiber-laser. Conventional process combines modified chemical vapor deposition (MCVD), to prepare porous silica frit of ultra high purity, with solution-doping approach. An active lasing element, such an erbium, is introduced into the laser material as an inorganic salt dissolved in an appropriate solvent [5]. Recent processing trends modify solution-doping approach or fully replace it, e.g. by aerosol deposition. Popular modification of solution-doping approach is based on the replacement of inorganic salts by inorganic nanoparticles. Such a simple modification can significantly improve properties of glass matrix [7], [8] thus improving the optical properties of optical fibers. However, the commercially available inorganic nanoparticles and nanocrystals usually don't meet the properties necessary to incorporate them into an optical fiber. The main requirements are placed on the composition of incorporated nanocrys-

tals, their size and chemical activity during the thermal processing of the optical fiber. Incorporated nanoparticles must be thermally stable above 2000 °C, their composition must appear suitable for luminescence properties and their size and shape must be below the scattering limits of the propagated waves. Suitable method to prepare ceramic nanocrystals with required properties is a photo-induced synthesis [9], [10]. This method allows to prepare ceramic nanocrystals with required properties. Moreover, introduced method can be readily combined with common fiber-optic technology.

In this paper, we present a versatile method providing an erbium-doped fiber distributed inside a lattice of nanocrystalline yttrium aluminium garnet. Conventional MCVD method was combined with a solution-doping deposition of ceramic nanocrystals of erbium-doped yttrium aluminium garnet which were prepared by photo-induced synthesis. Optical and waveguiding properties of prepared fiber were determined. Lasing properties of prepared fiber were proofed in a fiber-ring set-up. Achieved results were compared with properties of fibers prepared by a conventional solution doping method.

# 2. Experimental

## 2.1. Materials and Sample Preparation

Ceramic nanocrystals of erbium-doped yttrium aluminium garnet were prepared by photo-induced synthesis according to the published procedures [9], [10]. Concentration of erbium ions in YAG was 0.5 at %. To form a colloidal solution a total of 2 g of prepared nanocrystalline powder was dispersed in 200 ml of ethanol (UV-grade, Sigma-Aldrich). The dispersion was sonicated for 2 hours and filtered through a Millipore membrane of the porosity 0.4 μm. Prepared colloidal solution was long-term stable without sedimentation allowing a soaking into a porous frit.

Optical fibers were prepared by drawing from preforms manufactured by MCVD method combined with a solution-doping technique [5]. The process of doping was as follows: first, the porous silica frit of the length 300 mm was deposited at 1450 °C (by MCVD method) in the inner side of starting silica tube with a diameter 18 mm and a wall thickness 1.4 mm. The bubbler containing $SiCl_4$ was tempered to 25.0 °C and the flow of the carrying gas through the bubbler was 400 sccm. Then the silica frit was soaked with the colloidal solution of prepared nanocrystals for 24 hours. Then the colloidal solution was poured off the frit and the frit was dried in the oxygen flow 10 sccm for 48 hours followed by a thermal treatment at 600 °C to burn-out remaining organic matter. Afterwards the doped silica frit was sintered at 1400 °C and collapsed at 2100 °C into final preform. The preform was drawn into optical fiber with the outer diameter of 125 μm that was coated with a UV-curable acrylate (UV-A 3471, DeSoto).

## 2.2. Characterization Techniques

A refractive index profile of preform was measured on Photon Kinetics's A2600 refractive index analyzer. The local chemical composition of the preforms was determined by a Cameca SX100 electron microprobe. The samples were excited by the X-ray radiation beam of the energy equal to 15 keV. Beam current was 60 mA. A thin carbon layer was sputtered on the samples prior to analysis to prevent charging.

The angular distribution of output rays of the optical fiber was measured by the custom-made apparatus provided by Safibra s.r.o. The front face of analyzed fiber was placed into the 3-D micro-positioner that was preciously arranged into rotational axis of the goniometric cantilever with fixed laser diode emitting at 632 nm. The end-face of analyzed fiber was fixed inside the photo-detector. Cantilever rotates around the rotational axis varying the angle of incidence of light illuminating the front face of the fiber. Detected signal is recorded as a function of the intensity on the angle of incidence of the rotating laser diode.

Spectral attenuation and steady-state luminescence properties of the fiber were recorded on Ando AQ-6315B spectral analyzer. Longitudinal homogeneity of the fiber and background losses at 1310 nm were measured on Optical Time-Domain Reflectometer (OTDR) EXFO 715B. The width of the excitation pulse was 5 ns, the integration time was 30 s. Analyzed fiber was connected to spectrometer through the standard single-mode optical fiber.

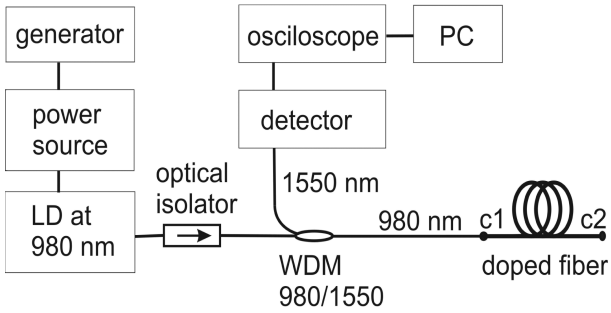


**Fig. 1:** Diagram of a cut-back method to measure a time-resolve luminescence of the fiber.

The time-resolved luminescence properties of the fiber were measured by the cut-back method. The experimental set-up is depicted in the Fig. 1. The

excitation laser diode EM4 (PowerNetix), operating at 976 nm with tunable power up to 250 mW, was controlled by a pulse-generator (Agilent). Emitted light was guided through the wavelength division multiplexer (WDM) to the analyzed fiber. The WDM were operating at 980 nm and 1550 nm and acted as a wavelength selection filter. The luminescence signal emitted at 1550 nm was demultiplexed and detected by the ultra-fast photo-diode. The time-response of the photo-diode was further analyzed by the 1 GSa·s$^{-1}$ oscilloscope (Agilent) synchronously with the leading pulses of the pulse-generator. The time-resolved spectra were recorded for different powers of the laser diode and different lengths of analyzed fiber to compensate the time delay introduced by the experimental set-up and by the wave-guiding properties of the analyzed fiber.

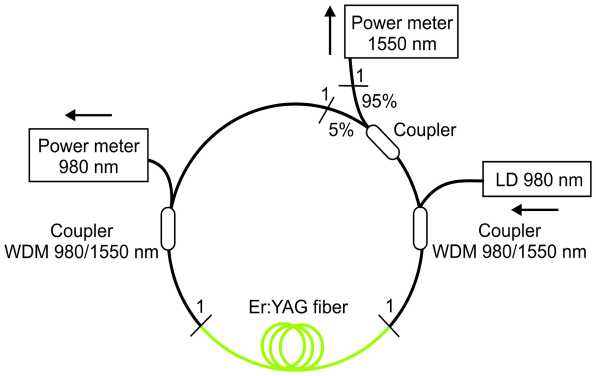


**Fig. 2:** Set-up of the fiber-ring laser.

The lasing properties of prepared fiber were measured in the ring arrangement [11] which is depicted in the Fig. 2. Segments of drawn fiber were spliced into a fiber ring consisting of two WDM operating at 980 nm and 1550 nm and a coupler splitting in the ratios 5/95 %. The active fibers were pumped by a laser diode EM4 (PowerNetix) operating at 976 nm with tunable power up to 250 mW. The intensities of the unabsorbed pumping signal at 980 nm and the laser signal at 1550 nm were measured by a dual-channel optical multimeter AQ2140 (Ando). Acquired data were displayed and processed in the Origin 8.0 software.

# 3. Results and Discussion

## 3.1. Characterization of Preform

Combination of the MCVD technology with the solution-doping of ceramic nanocrystals resulted into the formation of highly transparent preform. Refractive index profile had a gradient shape as can be seen in the Fig. 3. Maximal difference of refractive indexes between the preform core and preform cladding was 0.0092. The refractive index profile followed the concentration profile of the dopants inside the fiber core. The concentration ratios between particular dopants

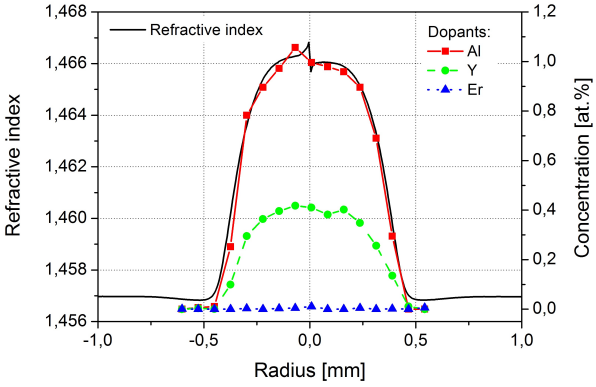


**Fig. 3:** Refractive index profile (left axis) and corresponding concentration profile of dopants (right axis).

corresponded to the composition of incorporated ceramic nanocrystals. Naturally, the concentrations of dopants in the preform were significantly lower due to the presence of the silica inside the preform core. Thus the maximal concentration of the aluminium ions was about 1 at % and corresponding concentration of erbium ions was 0.011 at % only. Such a value was up to ten times lower than the concentration or erbium ions in the fibers prepared by the conventional solution-doping methods [11], [12], [13] or in the fibers doped by commercially available erbium-doped alumina nanoparticles [7], [8]. The reason should be found in different sizes of dopants. Although, the primary size of prepared YAG's nanocrystals was smaller than 30 nm [10] the nanocrystals formed aggregates with the average size around 300 nm as had been proved by a dynamic-light scattering analysis. Such a size has limited the adsorption into the porous silica frit which typically appears the pore size ranging from 100 to 500 nm [14]. Conventional solution-doping method employs ionic solutions of dopants. Naturally, ions penetrates better even into small pores, thus the concentration of dopants inside the silica frit is significantly higher than in the case of deposition of colloidal nanocrystals which cannot penetrate into small pores due to their large size. As has been observed, deposition of colloidal nanocrystals improves the homogeneity of the collapsed preforms [7]. According to this observation the preform core appeared an excellent homogeneity comparing to the ytterbium-doped YAG which were prepared by conventional solution doping method [13] or by powder sinter technology [15].

## 3.2. Optical Properties of the Fiber

Prepared preform was successfully drawn into optical fiber. Shape and dimensions of the drawn optical fiber

were analyzed by optical microscopy. Outer diameter of drawn optical fiber was $125 \pm 2$ µm as can be seen in the Fig. 4. The size of the fiber core was 11 µm. Numerical aperture of optical fibers can be expressed by the equation [16]:

$$NA = \sqrt{n_{core}^2 - n_{cladding}^2}, \tag{1}$$

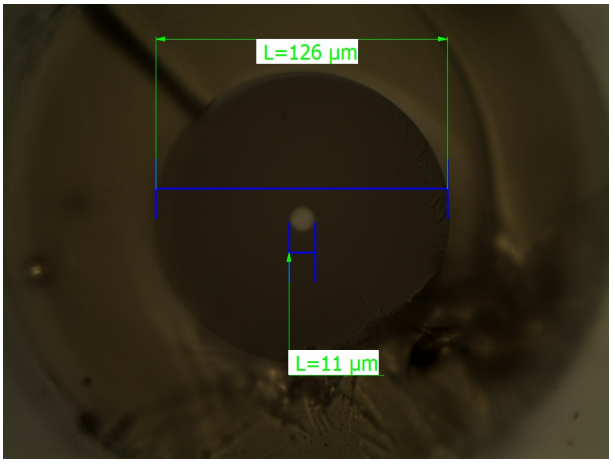


**Fig. 4:** Cross-section of drawn fiber visualized by optical microscopy.

where $n_{core}$ is the refractive index of the core of the fiber, $n_{cladding}$ is the refractive index of the silica cladding which is equal to 1.457. The numerical aperture was calculated from the refractive index profile of the preform, reaching the maximal value 1.4662, and verified by a measurement of angular distribution of optical fiber. The angular distribution of the fiber is demonstrated in the Fig. 5.

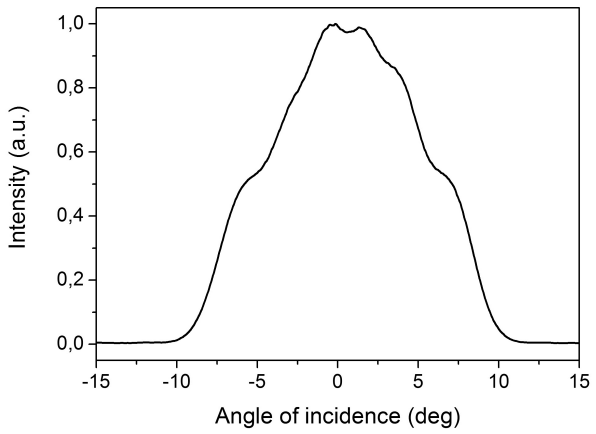


**Fig. 5:** Angular distribution of drawn fiber.

The value of the numerical aperture estimated for the 5 % limit of the maximal intensity was 0.167. The numerical aperture calculated according to the Eq. (1) was 0.164. Both values were close enough and the difference between them was within the error of the measurement. Numerical aperture together with the fiber-core size provided the information that drawn fiber has a character of a single-mode fiber.

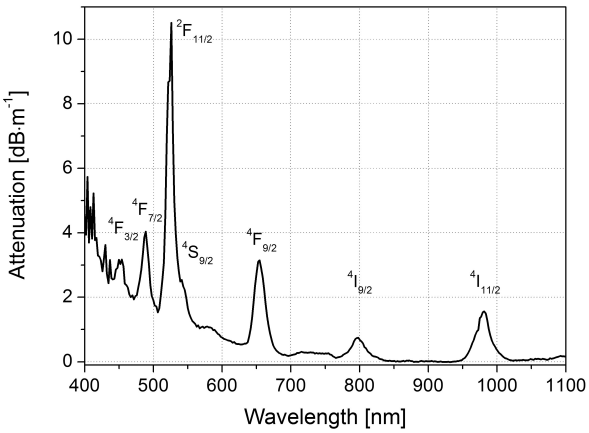


**Fig. 6:** Absorption spectrum of drawn fiber.

Attenuation of optical fibers can be divided into intrinsic and extrinsic components [16]. The intrinsic attenuation is given by optical properties of core material, i.e. silica glass. It covers material absorption, chromatic dispersion, scattering phenomena etc. It corresponds to the background losses of optical fibers. Recently prepared telecommunication optical fibers usually reach a theoretical value 0.4 dB·km$^{-1}$ [5]. Rare-earth doped optical fibers usually appear a background losses on the level of 0.05–0.5 dB·m$^{-1}$. The reason should be found in the formation of local fluctuations inside the glass structure which increase the scattering effects. The extrinsic attenuation is caused by the absorption of dopants and impurities presented in the fibers. Because of the MCVD technology, including the solution-doping modification, provides extremely pure materials, extrinsic attenuation practically corresponds to the absorption of introduced dopants and free –OH groups remaining in the silica glass matrix. Spectral attenuation of prepared fiber is depicted in the Fig. 6. Observed peaks had the shape and the positions typical for erbium-doped optical fibers. Background losses recorded at 850 nm and 1310 nm were 0.021 dB·m$^{-1}$ and 0.136 dB·m$^{-1}$ respectively. The attenuation at 1310 nm was slightly higher because this spectral region overlaps the absorption peak of free –OH groups.

Emission spectrum of the fiber excited at 976 nm by a laser diode is demonstrated in the Fig. 8. The overall shape of the emission band is similar to that of the fibers prepared by conventional solution doping methods [5], [7]. Intensity of the emission linearly increased with increasing excitation power. A lifetime of a transition $^4I_{13/2} \longrightarrow {}^4I_{15/2}$ was $10.2 \pm 0.1$ ms.

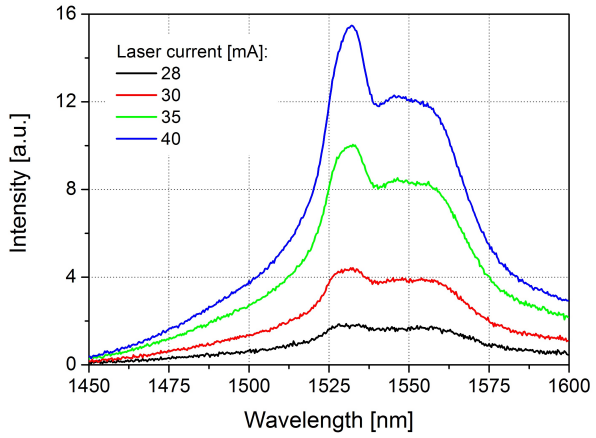


**Fig. 7:** Emission spectrum of drawn fiber excited at 976 nm.

This value is slightly higher than a lifetime of erbium ions distributed inside alumina co-doped optical fibers [7]. Moreover, it well matches the value 10.98 ms that was predicted for polycrystalline Er-doped YAG [17]. The slight reduction of the lifetime corresponds to the observation achieved on Er-doped YAG ceramic nanoparticles [18]. It was found that the smaller size of nanoparticles decreases the quantum yield of the luminescence due to the high concentration of surface defects in the nanocrystalline matrix.

### 3.3. Characterization of the Fiber-Laser

The prepared fiber was tested as a gain medium in a fiber ring laser. The prepared fiber was spliced in the ring and the dependence of the output laser power ($P^{output}$) on the power of the pumping laser ($P^{pump}$) was measured. The tested fiber was successively shortened to found out the optimal fiber length. The results are demonstrated in the Fig. 8.

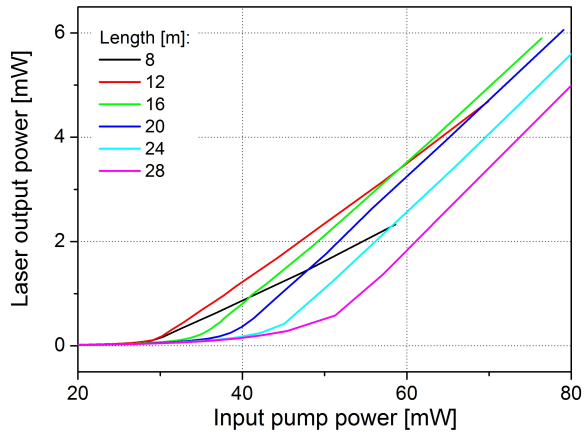


**Fig. 8:** Power conversion in fiber-laser set-up.

A limit when a stimulated emission overweighs a spontaneous emission is defined as a laser threshold. Laser thresholds for particular fiber lengths were calculated as the intercepts of linear fits from the dependence of output laser power on the power of the pumping laser. Laser thresholds linearly rose up with the length of inserted fiber. The lowest value 28.8 mW was reached for the fiber length equal to 12 m, maximal measured value equal to 47.9 mW was reached for the fiber length of 28 m.

A slope efficiency $\eta$ is a characteristic parameter of the laser which is defined as a slope of the ratio of output laser power ($P^{output}$) over the power of the pumping laser ($P^{pump}$) according to the Eq. (2):

$$\eta = \frac{dP^{output}}{P^{pump}}. \tag{2}$$

As can be seen in the Fig. 9, the slope efficiency of the fiber laser rose up with increasing length of the active fiber. The efficiency practically reached the saturation around 15.4 % for the fiber longer than 20 m. Considering an optimal performance of the fiber laser, the requirements are placed on high slope efficiency, low threshold power of the laser and short lengths of employed fiber. These requirements were fulfilled for the fiber length of 20 m.

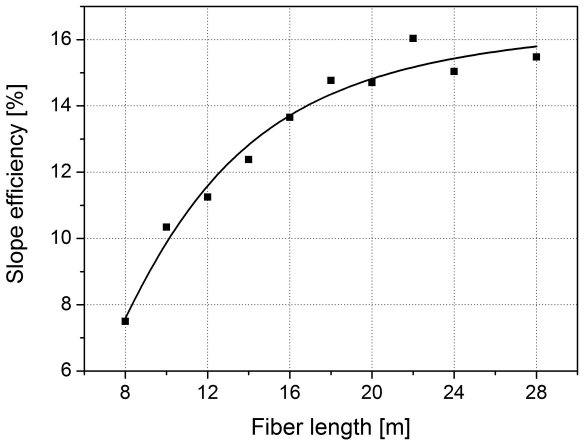


**Fig. 9:** Slope efficiency of fiber laser for different lengths of active fibers.

The properties of prepared fiber were compared with the properties of optical fiber prepared by conventional solution-doping method. The optimal parameters reached for the erbium-doped fiber prepared by conventional solution doping method were following [19]: fiber length 5 m, slope efficiency 23 %. Despite these parameters are better than presented results, it should be noted, that the concentration of erbium ions in the referred fiber was roughly ten times higher. It can be expected that the increase of the concentration of dopants can improve the laser properties of the

fiber prepared by the deposition of ceramic nanocrystals. Such a step can be realized by several technological improvements. First one is an increase of erbium ions in the incorporated powder. The second is the grinding of the powder to smaller particle size to allow their penetration even in small pores of the frit. The third improvement is the modification of the silica frit by another dopant. Co-doping during the MCVD process can improve the pore-size distribution of the silica frit as was described elsewhere [14].

## 4. Conclusion

Erbium-doped active optical fiber was successfully prepared by incorporation of ceramic nanocrystals inside a core of optical fiber. Novel method combining the common MCVD process with the solution-doping of Er-doped yttrium aluminium garnet nanocrystals was successfully tested. Active single-mode erbium-doped fiber was successfully prepared by presented approach. Prepared fiber showed the luminescence properties typical for trivalent erbium ions distributed inside nanocrystalline lattice. The lifetime of the transition $^4I_{13/2} \longrightarrow {}^4I_{15/2}$ was 10.2 ms. Lasing properties of prepared fiber were demonstrated in a ring laser set-up. Optimal laser properties were achieved for a fiber length of 20 m. Corresponding laser threshold was 37 mW. The slope efficiency of the fiber-laser was about 15 %. Presented approach can be simply extended to the deposition of other ceramic nanomaterials.

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