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Optical properties of Nd³⁺-doped silica fibers obtained by sol-gel method

W. Strek^{a,*}, E. Pawlik^b, P. Dereń^c, A. Bednarkiewicz^c, J. Wojcik^d, V.E. Gaishun^e,
G.I. Malashkevich^{b,e}

^a*Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Wroclaw, Poland*

^b*Institute of Acoustics and Telecommunication, Technical University of Wroclaw, Wroclaw, Poland*

^c*Department of Optical Fiber Technology, Maria Curie-Skłodowska University, Lublin, Poland*

^d*Advanced Materials Research Laboratory, Gomel State University, Gomel, Belarus*

^e*Institute of Molecular and Atomic Physics, National Academy of Sciences of Belarus, Minsk, Belarus*

Abstract

The Nd³⁺-doped sol-gel silica fiber of clad-polymer type, 125 μm in diameter, was pulled from the preform with concentration of active ions of 0.03 mol.%. The optical properties of such a fiber were investigated and compared to monolithic samples. The attenuation factor for this fiber was determined at 1.08 μm to be 90 dB/km. The difference in emission spectra between neodymium-doped sol-gel silica bulk samples and fibers manifesting in a significant shifting of the peak emission of ⁴F_{3/2} → ⁴I_{11/2} transition was observed. The fluorescence of Nd³⁺ silica gel fiber was measured as a function of length of fiber and excitation power. It was found that the fluorescence intensities and lifetimes were slightly dependent on fiber length. Moreover it was noted that the intensity of fluorescence increased linearly with power. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Optical properties; Fiber length; Excitation power; Fluorescence intensity

1. Introduction

Among the rare earth ions much attention has been given to research and development of Nd³⁺ (1.05 μm), Pr³⁺ (1.3 μm) and Er³⁺ (1.5 μm) doped glass fibers for telecommunication systems. This interest is associated with excellent transmission parameters in the telecommunication regions of silica low-loss windows. Among the different practical solutions of optoelectronics structures appropriate for construction of rare earth doped amplifiers, the glass planar lightguides and the glass fibers most often find application. For manufacturing optical fibers the most popular is the fluoride glass ZBLAN [1] which is characterised by low energy vibrations leading in consequence to reduced nonradiative transitions. Another critical parameter limiting optical efficiency is clustering of lanthanide ions and contamination of hydroxyl groups. The first limits, sometimes, not only optical performance of erbium-doped fiber amplifier (EDFA) by several orders of magnitude but leads also to appearance of an up-conversion effect (green emission). To avoid aggregation of metal ions the active glass is co-doped with aluminium ions, however,

the synthesis of such glasses is difficult to control. The second one leads to enhancement of multiphonon relaxation and attenuation factor in the regions of OH⁻ overtones.

Recently it was discovered that the sol-gel technology [2] allows to manufacture the erbium silica glasses co-doped with aluminium, titanium or germanium ions with greatly reduced contamination of hydroxyl groups. The optical properties of erbium-doped silica gel fibers were reported earlier by us [3,4]. The low-loss erbium-doped single mode sol-gel fibers were reported by Wu et al. [5]. The sol-gel technique was also applied for fabrication of silica optical fibers co-doped with Er and Yb ions [6].

The optical properties of Nd³⁺-doped silica glass obtained by sol-gel method were earlier described by Pope and Mackenzie [7] and Fujiyama et al. [8]. The effect of aluminium ions on clustering processes in the Nd³⁺-doped silica sol-gel glasses were reported by Malashkevich et al. [9].

In this paper we report the optical properties of Nd³⁺ ion in silica sol-gel fiber. It was found that the emission spectra and decay times of Nd³⁺ were slightly dependent on length of fiber. The maximum of ⁴F_{3/2} → ⁴I_{11/2} transitions was shifted in red to 1.09 μm compared to the bulk

*Corresponding author.

preform where it was 1.05 μm . Also the branching ratios were significantly different. The effect of excitation power on emission characteristics was also investigated.

2. Experimental

The monolithic sol-gel silica samples doped with Nd^{3+} ions were manufactured according to the impregnation method described by us earlier [10]. The fibers were pulled from the small rods of Nd^{3+} doped silica preforms into the PCS-type fiber with the core diameter of 125 μm . The Nd^{3+} concentration was determined to be 300 ppm.

The absorption spectrum was measured on an Ocean Optics SD2000 spectrophotometer. Luminescence spectra were measured on a Jobin-Yvon spectrophotometer TRW 1000 using an argon laser as excitation source. Emission lifetimes were measured with Tektronics 1000 TDS 380 oscilloscope. The attenuation of Nd^{3+} -doped fiber was measured by means of the cut-off technique [11].

3. Results and discussion

3.1. Optical properties of Nd^{3+} -doped silica bulk samples

The absorption spectrum of a monolithic sample of silica gel glass doped with Nd^{3+} ions is shown in Fig. 1. The absorption spectra were measured for different concentrations of Nd^{3+} ions at room temperature but we did not observe any significant changes with concentration.

The assignment of f–f transitions of Nd^{3+} ion is given in the figure. The refractive index of the sample was determined to be 1.4570.

Emission spectra of monolithic samples of Nd^{3+} -doped silica gel glasses are shown in Fig. 2. The emission originates from the ${}^3\text{F}_{3/2}$ level. The observed bands were attributed to the ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{9/2}$ and ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{11/2}$ transitions. The emission spectra were measured for several concentrations of Nd^{3+} ions. The intensity branching ratio defined as

$$\beta_{11/2} = I({}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{11/2}) / I({}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{9/2} + {}^4\text{I}_{11/2})$$

was measured to be 0.39.

The measured emission decay curves were found to be perfectly exponential and the fluorescence lifetime of Nd^{3+} -doped silica glass host was determined to be 500 μs . It was somewhat higher than the fluorescence lifetimes measured earlier by us for the Nd^{3+} -Al-co-doped SiO_2 glasses [9].

3.2. Optical properties of Nd^{3+} -doped silica fiber

The attenuation of Nd^{3+} -doped fiber was measured in the optical range 400 to 1200 nm (see Fig. 3) for a fiber with concentration of 0.03 mol.%. In the region of 1.08 μm the loss factor was determined to be not higher than 90 dB/km.

The emission spectra of Nd^{3+} -doped silica gel fiber with a nominal concentration of 0.03 mol.% were excited by means of argon laser. The spectra are shown in Fig. 4. They are essentially similar to those measured for a

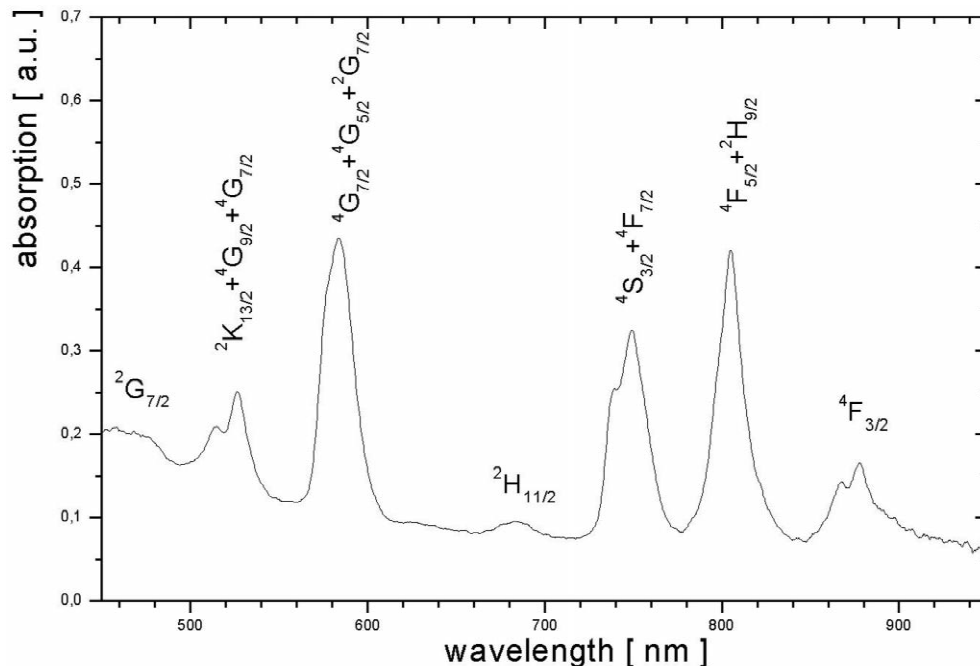


Fig. 1. Absorption spectrum of Nd^{3+} -doped silica sol-gel glass.

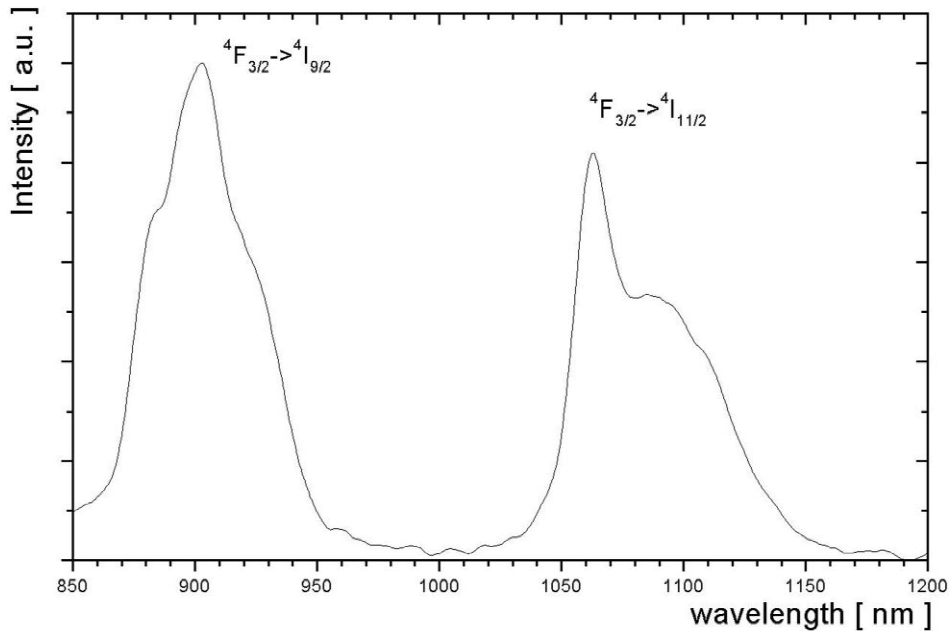


Fig. 2. Emission spectra of Nd^{3+} -doped silica sol-gel glass measured for different concentration of active ions.

preforma host glass. Especially the resonant transition ${}^4F_{3/2} \rightarrow {}^4I_{9/2}$ demonstrated increasing intensity with the fiber length. The calculated intensity branching ratio $\beta_{11/2}$ was determined to be 0.27. It is much lower than that one measured for a bulk sample. It is interesting to note that the intensity peak of the laser transition ${}^4F_{3/2} \rightarrow {}^4I_{11/2}$ at 1.09 μm was shifted to the red compared to other Nd^{3+} glass hosts (see Table 1).

The fluorescence lifetimes of Nd^{3+} ion in silica sol-gel

fiber were measured for different lengths of fibers. The decay curves were exponential. We have noted that the fluorescence lifetimes were slightly dependent on fiber lengths. The measured values are listed in Table 1. The emission lifetimes for the fiber length 7 m was determined to be 450 μs . It was slightly higher than that one measured for a Nd^{3+} -Al-co-doped SiO_2 fiber – 400 μs [12].

The emission spectra of Nd^{3+} -doped silica fiber were also measured versus excitation power. The effect of

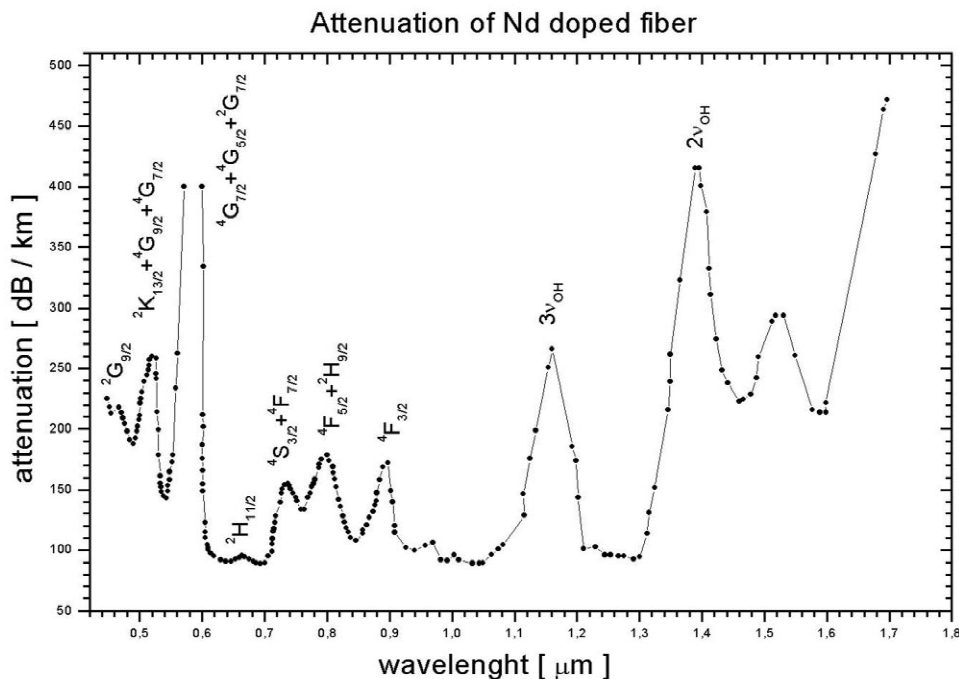


Fig. 3. Attenuation of Nd^{3+} -doped silica gel fiber versus wavelength for concentration 0.03 mol.%.

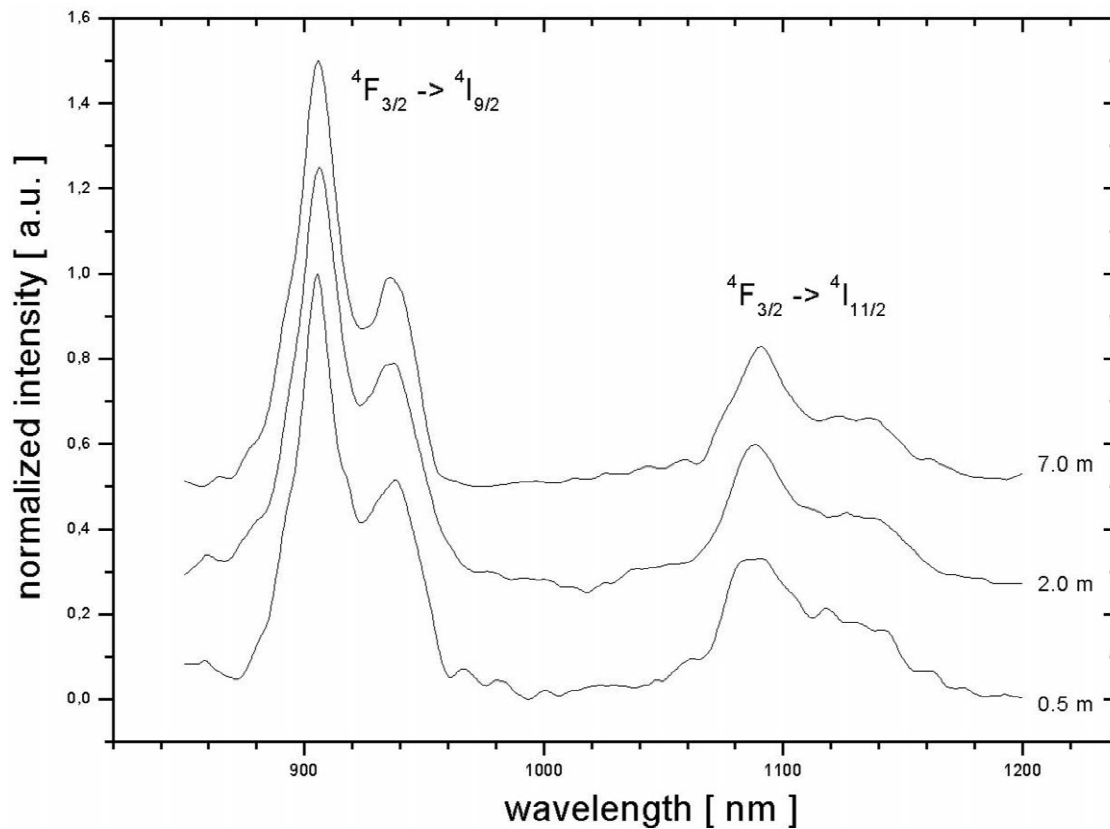


Fig. 4. Emission spectra of Nd^{3+} -doped silica gel fiber versus fiber length excited by 514 nm argon laser.

excitation intensity on Nd^{3+} emission in silica sol-gel fiber is shown in Fig. 5. We have found that the intensity of Nd^{3+} emission was not dependent on excitation power. One can also note that there is no change in emission profile.

4. Conclusions

The Nd^{3+} -doped silica gel glass in the form of bulk samples with a small contamination of hydroxyl groups were fabricated. The bulk preform with a low concentration of neodymium ion was then used for pulling the

optical fibers. The absorption and emission properties of Nd^{3+} ion were investigated in bulk samples and compared to those measured in the fiber. We have found that the emission characteristics of the fiber were significantly different to those measured for a bulk sample. The emission peak of the ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{9/2}$ transition measured at $1.09 \mu\text{m}$ was significantly shifted into the red compared to the bulk. The effect of excitation power on emission spectra was investigated. It was found that the emission intensity increased linearly with excitation pumping. It means that the co-operative processes associated with the excited state absorption and the energy transfer play a minor role. The obtained results allow us to conclude that

Table 1
Characteristics of Nd^{3+} -doped glasses (bulk and fibers)

Glass composition	Nd^{3+} conc. (ppm)	Wavelength (nm)	Branching ratio ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_J$		Notes
			$J = 9/2$	$J = 11/2$	
$\text{Nd}^{3+}:\text{SiO}_2(100\%)$ fiber		1088	0.59	0.41	[1]
$\text{Nd}^{3+}:\text{SiO}_2(11\%)/\text{BaO}$ (5%) fiber		1067	0.48	0.52	[1]
$\text{Nd}^{3+}:\text{SiO}_2$ (bulk)	2500	1064	0.61	0.39	$\tau = 543 \mu\text{s}$
$\text{Nd}^{3+}:\text{SiO}_2$ (fiber)	300	1088	0.73	0.27	$\tau = 488 \mu\text{s}$

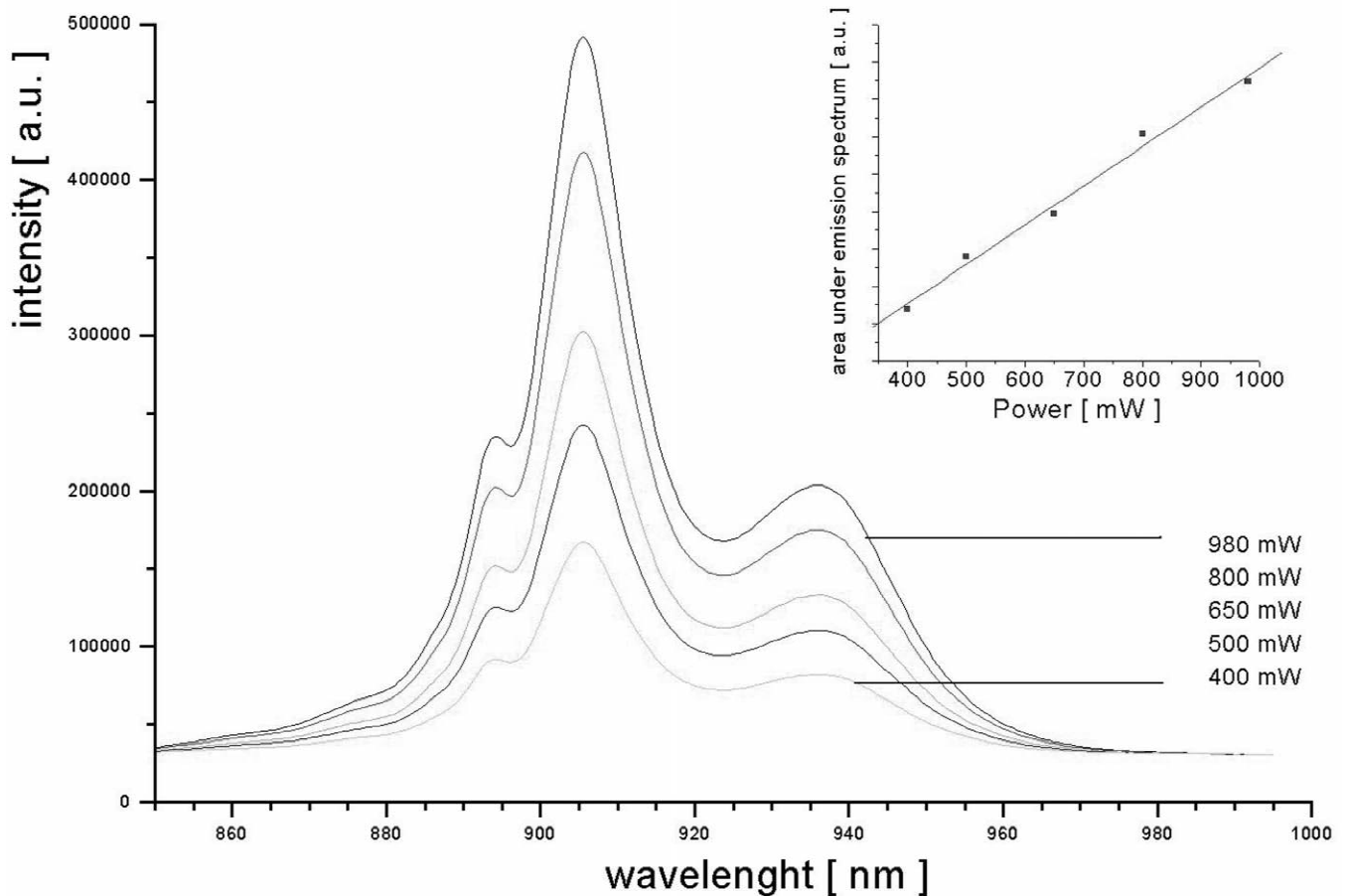


Fig. 5. Power dependence of emission intensity of Nd^{3+} -doped silica sol-gel fiber.

the quality of manufactured silica sol-gel fiber is quite appropriate for construction of optical amplifiers operating at the range of $1.09 \mu\text{m}$.

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