Study of the luminescence properties of $Nd(TTA)_3$phen-doped 6-FDA/epoxy waveguides

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ABSTRACT

Fluorinated Epoxy waveguides doped with Nd complexes have been studied for optical amplification applications. The fluorescent complex was $Nd(TTA)_3$phen (TTA = thenoyltrifluoroacetone, phen = 1, 10-phenanthroline), which was mixed with the host material 6-FDA (6-fluorinated-dianhydride). The solution was spin coated in order to obtain $Nd(TTA)_3$phen-doped 6-FDA/epoxy slab and channel waveguides. The emission spectra of the Nd complex doped waveguides were measured at different pump powers by pumping at 800nm, and emission was observed at 890nm, 1060nm and 1330nm. The luminescence lifetime of the Nd complex within the waveguides was experimentally determined. The results demonstrate that the neodymium ions within the polymer host have good transition properties. Based on experimentally obtained parameters the optical gain of the Nd complex doped waveguides was estimated with the aid of rate equations. The results show that Nd complex doped polymer waveguides are promising gain media for optical amplification.

Keywords: Nd complex, polymer waveguides, fabrication, emission spectrum, luminescence lifetime, small signal gain

1. INTRODUCTION

Lasing and amplification by rare-earth doped polymer materials have been widely investigated. Polymers are promising host candidates for these applications because of their excellent properties such as high transparency, low cost, and easy fabrication. Recently, work has been done on rare-earth-doped polymer planar waveguides [1-4].

In the following we describe our work on Nd-doped polymer waveguides. For this, $Nd(TTA)_3$phen was synthesized and doped into the fluorinated host 6-FDA epoxy. The ligands with fluorine and the fluorination of the host material can improve the luminescence quenching from C-H and water.

The $Nd(TTA)_3$phen-doped 6-FDA/epoxy film was spin-coated on an thermally oxidized wafer. The room-temperature absorption spectrum of neodymium ions in 6-FDA/epoxy slab waveguide was experimentally determined in order to estimate with the aid of the Judd-Ofelt theory the transition properties of neodymium ions in polymer materials. Also the spontaneous emission spectrum of the $Nd(TTA)_3$phen-doped 6-FDA/epoxy slab waveguide was obtained with three peaks at 890nm, 1060nm and 1330nm. The spectroscopic results of Nd-doped slab waveguides indicate this Nd-complex doped polymer material is promising to be used in planar waveguide lasers and amplifiers.

For this kind of application, confinement of the light in two dimensions is crucial. Therefore, we continued our work with Nd-complex doped channel waveguides. $Nd(TTA)_3$phen-doped 6-FDA/epoxy channel waveguides with cross section of $40\mu m \times 40\mu m$ were fabricated by backfilling the core material in inverted cladding channels. The emission spectrum of the Nd-doped channel waveguide was measured at different pump powers. The result provides evidence that stimulated emission occurs at 1060nm and 1330nm.

Luminescence lifetimes of neodymium ions within the polymer host were experimentally measured and the results were promising.

With the experimental results and rate equations, a theoretical estimate is given of the optical gain of our Nd complex doped waveguides.

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2. STUDY OF NEODYMIUM DOPED SLAB WAVEGUIDES

2.1 Fabrication of Nd-doped polymer slab waveguides

Polymers are usually poor host materials for rare-earth ions in optical amplifications due to the presence of high-energy vibrations from C-H bonds and water. One way to improve the luminescence quenching in rare-earth doped polymer host is to encapsulate the ions in organic chelates and dope these complexes directly.

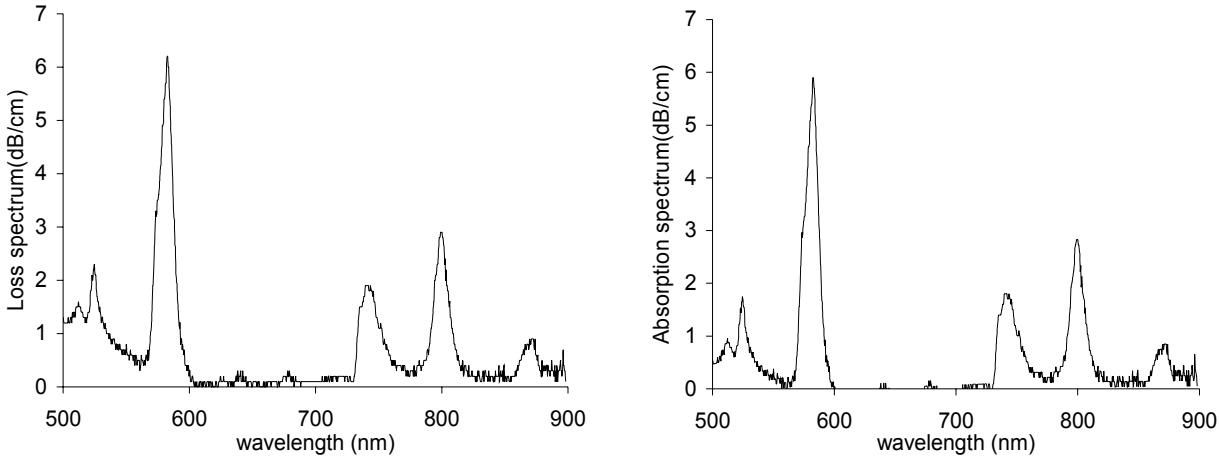
In our work, the $Nd(TTA)_3phen$ was synthesized according to the procedure as described in [5] and doped into 6FDA/epoxy. The ligands with fluorine can reduce the luminescent quenching from C-H. The host material with epoxy groups can displace water that is associated to a complex and, therefore, will reduce the energy vibration from water.

A film of $Nd(TTA)_3phen$-doped 6-FDA epoxy was spin-coated on an thermally oxidized wafer. The refractive index and the thickness as determined with a prism coupler were 1.53 (at 632.5nm) and 4.3 μm, respectively. The concentration of neodymium ions was 1.1×10^{19} Nd/cm^3.

2.2 Loss spectrum and absorption spectrum

White light was coupled into the film and coupled out after propagating a certain distance through the film using a prismcoupler. Varying the propagation distance and collecting the light from the outcoupling prism by a spectrum analyzer (Spectro320, Instrument System), the room-temperature loss spectrum of neodymium ions in 6-FDA epoxy was recorded (Fig. 1.a).

The absorption spectrum can be obtained with subtraction of the background loss from the recorded loss spectrum. The absorption spectrum of neodymium ions in 6-FDA epoxy in Fig. 1.b clearly shows the absorption peaks of neodymium ions at 580nm, 740nm, 800nm and 865nm.



(a) (b)

Fig. 1 Room temperature loss spectrum from 500 to 900 nm (a) of a Nd^{3+} in a 6-FDA/epoxy slab waveguide and (b) the same as (a) after subtraction of the loss spectrum of the undoped 6-FDA/epoxy host.

2.3 Judd-Ofelt analysis

Judd-Ofelt theory [6, 7] is a successful and widely used theory for characterization of 4*f* transitions in rare-earth doped materials. In our case the standard Judd-Ofelt analysis was applied for the study of the optical transitions in $Nd(TTA)_3phen$-doped 6-FDA/epoxy planar waveguide.

The electric dipole line strength *S* is introduced to describe the transition between two eigenstates of the ion in the Judd-Ofelt theory. The line strength *S* between initial state *J* characterized by $\left|(S,L)J\right\rangle$ and the final state *J′* given by $\left|(S',L')J'\right\rangle$ can be written as [8~11] (*J* is the angular momentum):

$$S_{calc}(J \to J') = \sum_{t=2,4,6} \Omega_t \left| \left\langle (S,L) J \left\| U^{(t)} \right\| (S',L') J' \right\rangle \right|^2 = \Omega_2 \cdot \left[U^{(2)} \right]^2 + \Omega_4 \cdot \left[U^{(4)} \right]^2 + \Omega_6 \cdot \left[U^{(6)} \right]^2 \quad (1)$$

where Ω_t (t=2,4,6) are the Judd-Ofelt parameters, and $U^{(t)}$ (t=2,4,6) are the doubly reduced matrix elements depending only on angular momentum The parameters Ω_t (t=2,4,6) can be used to calculate line strength between any pair of excited states. They are determined by measuring the line strength for a number of ground-state transitions.

Four absorption peaks can be observed from Fig. 1.b in the range from 500 to 900 nm, which allow to determine the line strengths $S_{meas}(J \to J')$ of the bands by using the following expression:

$$S_{meas}(J \to J') = \frac{3ch(2J+1)n}{8\pi^3 \lambda e^2 N_0} \left[\frac{9}{(n^2+2)^2} \right] \Gamma \quad (2)$$

where c is the velocity of light, h is the Planck's constant, e is the elementary charge, N_0 is the density of ions, λ is the mean wavelength of the absorption bands, n is the wavelength-dependent refractive index which is determined from Sellmeier's dispersion equation, and $\Gamma = \int \alpha(\lambda) d\lambda$ is the integrated absorption coefficient obtained from the absorption spectrum.

From a least-squares fit of S_{meas} to S_{calc}, the following values of the three parameters Ω_t (t=2,4,6) can be obtained:

$$\Omega_2 = 16.64 \times 10^{-20} cm^2, \Omega_4 = 8.85 \times 10^{-20} cm^2, \Omega_6 = 11.83 \times 10^{-20} cm^2$$

It has been shown that Ω_2 is quite sensitive to the metal-ion environment [12]. A large value of Ω_2 indicates that the optical transition is dominated by the electrical dipole transition in this system and the emission is strong. Compared with the values of the Judd-Ofelt parameters of neodymium ions in other hosts from literature [9], our Nd-doped material has a large Ω_2.

The radiative decay rate, radiative lifetime and fluorescence branching ratio can be obtained based on the Judd-Ofelt parameters [10] and are given in Table 1.

Table 1. Predicted radiative decay rates, radiative lifetimes, and branching ratios of Nd^{3+} in 6-FDA/epoxy slab waveguides at 300K.

Transition	Wavelength (nm)	$A_{JJ'}$ (ms^{-1})	τ_{rad} (μs)	$\beta_{JJ'}$
$^4F_{3/2} \rightarrow {}^4I_{9/2}$	865	1.9491	182.6	0.3559
$^4F_{3/2} \rightarrow {}^4I_{11/2}$	1060	2.9168		0.5326
$^4F_{3/2} \rightarrow {}^4I_{13/2}$	1330	0.5812		0.1061
$^4F_{3/2} \rightarrow {}^4I_{15/2}$	1330	0.0297		0.0054

The radiative lifetime τ_{rad} for neodymium in our material is comparable to that in deuterated (-d_8) PMMA, but smaller than the values in the hosts of PF (perfluorinated) plastic solution and ZBAN (ZrFr-BaF_2-LaF_2-ALF_3) [9]. The reason is that the latter two materials don't contain C-H bonds which cause quenching.

2.4 Emission spectrum

The absorption spectrum given in Fig. 1.b shows there is a strong absorption peak of neodymium ions around 800nm. For the emission measurements a Ti:Sapphire laser was tuned at 800nm as the excitation source. The spontaneous emission spectrum was recorded by the optical spectrum analyzer (Spectro 320) and is shown in Fig. 2. Three distinct emission bands with peaks at 890 nm, 1060nm and 1330nm can be seen clearly. They correspond to transitions from the $^4F_{3/2}$ level to the $^4I_{9/2}$, $^4I_{11/2}$, and $^4I_{13/2}$ levels. The spectrum indicates clearly that the neodymium ions in the waveguide are active.

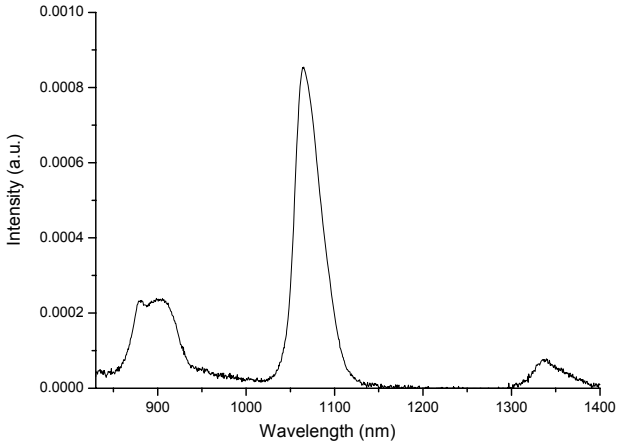


Fig. 2 Room temperature spontaneous emission of the Nd(TTA)3phen doped 6-FDA/epoxy film pumped by a Ti:Sapphire laser at 800nm.

3. STUDY OF NEODYMIUM DOPED CHANNEL WAVEGUIDE

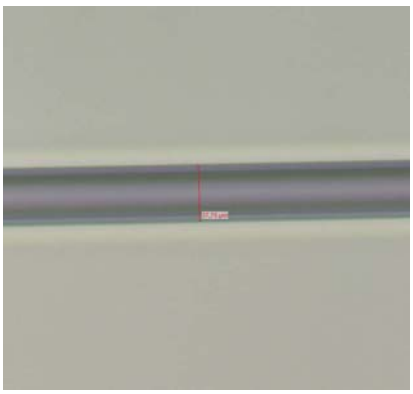
3.1 Channel waveguide fabrication

The study on Nd complex doped slab polymer waveguides shows that $Nd(TTA)_3phen$ doped 6-FDA/epoxy is well suited for lasing or amplification in optical waveguides. Therefore, we continued our study on Nd complex doped channel waveguides.

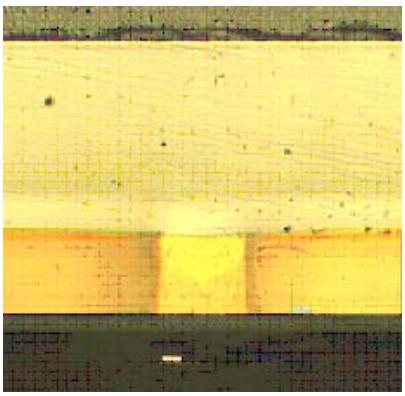
We use the cycloaliphatic epoxy prepolymer (code name CHEP) [13] as the cladding material of Nd-complex doped polymer channel waveguides. It is a photodefinable material with refractive index of 1.51. By spin-coating and photodefining CHEP, inverted channels with cross section of $40\mu m \times 40\mu m$ in the low index CHEP polymer were obtained on a thermally oxidized wafer.

The core material, a $Nd(TTA)_3phen$ doped 6-FDA/epoxy solution, was then backfilled via spin-coating twice and the Nd doped channel waveguide was realized after thermal curing. Fig. 3.a is the microscope picture of Nd-doped channel after backfilling the core material.

A lower refractive index silicon containing epoxy was used as the upper cladding after UV exposure, and on the top of which a Pyrex glass wafer was applied to improve the quality of dicing and protect the structure. Fig. 3.b shows a microscope picture of the cross section of the waveguide.



(a)



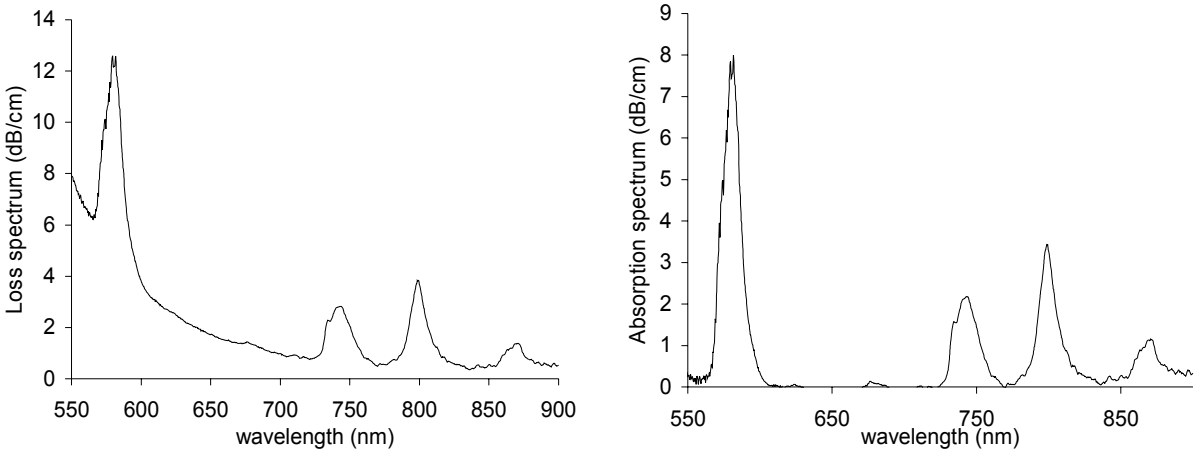
(b)

Fig 3 Microscope pictures of $Nd(TTA)_3phen$ doped 6-FDA/epoxy 40 x 40 μm^2 channel waveguides; (a) top view; (b) cross section.

3.2 Loss spectrum and absorption spectrum

The waveguide loss was determined with the cutback method. With a broadband white light source (FemtoPower1060, SC450, Fianium) at the input of the samples with different lengths, the optical output was coupled into a 50 μm diameter multimode fiber and collected by a spectrum analyzer. Fig. 4.a shows the resulting loss spectrum of $Nd(TTA)_3$phen-doped 6-FDA/epoxy channel waveguides.

The absorption spectrum (Fig. 4.b) obtained after subtraction of the background from loss spectrum clearly shows the absorption lines of the neodymium, which appear at 580, 740, 800 and 865 nm.



(a) (b)

Fig. 4 Room temperature spectrum of Nd^{3+} in 6-FDA/epoxy channel waveguide from 550nm to 900nm; (a): loss spectrum, (b): absorption spectrum obtained after subtraction of background from (a).

3.3 Judd-Ofelt analysis

The Judd-Ofelt parameters of neodymium ions in channel waveguide could be determined with the aid of the absorption spectrum as follows:

$$\Omega_2 = 25.27\times10^{-20}cm^2, \Omega_4 = 11.36\times10^{-20}cm^2, \Omega_6 = 14.36\times10^{-20}cm^2$$

With these parameters, radiative decay rates, radiative lifetimes, and branching ratios can be obtained and are presented in Table 2. The results show similar transition properties as that of neodymium ions in slab waveguides. The value of Ω_2 is even larger than of in the slab waveguide, which indicates a stronger emission in the channel waveguide.

Table 2. Predicted radiative decay rates, radiative lifetimes, and branching ratios of Nd^{3+} in 6-FDA/epoxy channel waveguide at 300K.

Transition	Wavelength (nm)	$A_{JJ'}$ (ms^{-1})	τ_{rad} (μs)	$\beta_{JJ'}$
$^4F_{3/2} \rightarrow {}^4I_{9/2}$	865	2.4696	147.2	0.3635
$^4F_{3/2} \rightarrow {}^4I_{11/2}$	1060	3.5825		0.5273
$^4F_{3/2} \rightarrow {}^4I_{13/2}$	1330	0.7053		0.1038
$^4F_{3/2} \rightarrow {}^4I_{15/2}$	1330	0.0361		0.0053

3.4 Emission spectrum and power dependent measurement

The infrared fluorescence of the Nd-complex doped channel waveguide was experimentally obtained by pumping at 800nm (Fig. 5).

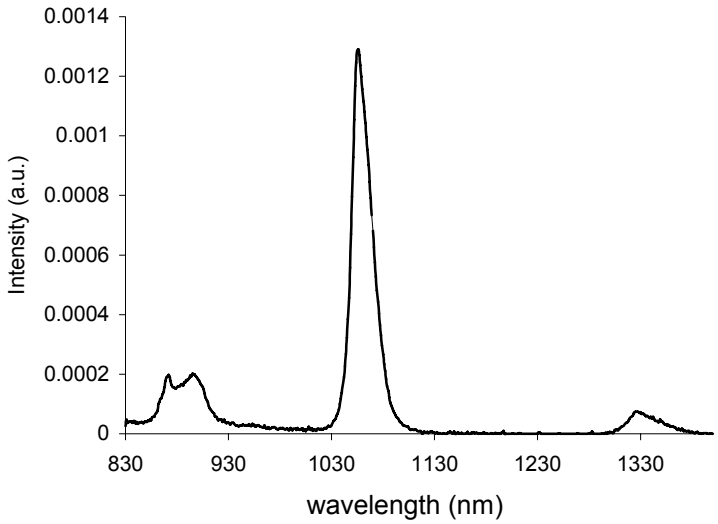
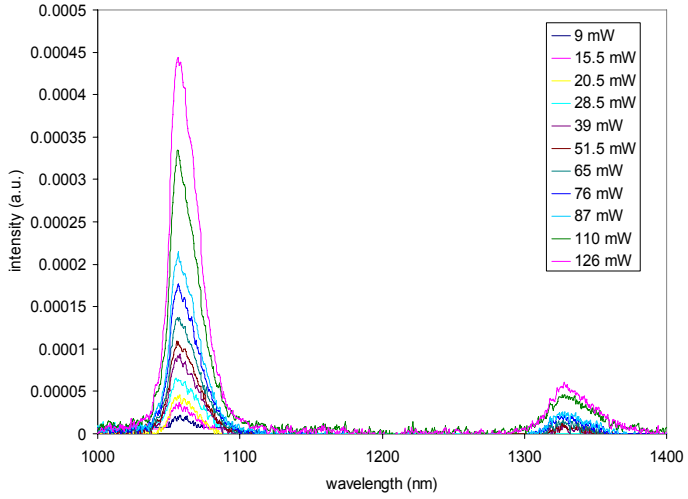


Fig. 5 Room temperature emission spectrum of a $Nd(TTA)_3Phen$ doped 6-FDA epoxy channel waveguide pumped by a Ti:Sapphire laser at 800nm.

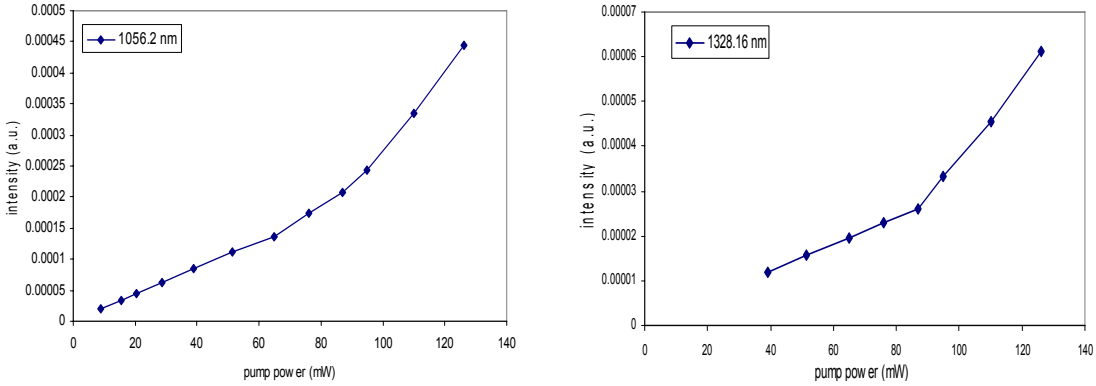
Further study was focused on the emission spectrum around 1060nm and 1330 nm with higher resolution. Fig. 6a shows the emission spectra of the Nd-doped channel waveguide at different pump powers. Fig. 6b and 6c give the intensity of the emission peaks (1056nm and 1328nm) as the function of pump power.

The emission intensity as a function of pump power shows a clear nonlinear behavior when the pomp power exceeds 50mW. The slope of the first three points can be linearly extrapolated, as expected, to the origin with zero intensity at zero pump power. The value of the slope of the rest of the points at higher pump powers in Fig. 6b and 6c is about three time of the value of the slope at lower pump powers.

This nonlinear response observed in the emission spectrum at high pump powers with much larger pumping efficiency is an indication of stimulated emission at 1060nm and 1330 nm in this Nd-complex doped sample. In literature, similar results have been reported from pump power dependent measurements with similar materials and were attributed to amplified spontaneous emission (ASE) [14, 15].



(a)



(b) (c)

Fig. 6 (a) The emission spectra of a Nd-doped channel waveguide with different input powers; (b) The intensity of the maximum of the emission spectrum at 1059nm as function of input power; (c) The intensity of the maximum of the emission spectrum at 1330nm as function of input power.

4. LUMINESCENCE LIFETIME MEASUREMENT

The lifetime of the metastable state can reveal information such as the quenching due to the interaction between the neodymium ions and their surroundings. In our $Nd(TTA)_3$phen-doped 6FDA/epoxy, ligands with fluorine and fluorination of the host material were used to reduce the luminescence quenching from C-H bonds and water. Besides that, we replaced the water molecules of the Nd chelate with phen (1, 10-phenanthroline) to reduce the high-energy H-O vibration of H_2O.

4.1 Experimental setup for the determination of the luminescence lifetime

The experimental setup for the determination of the luminescence lifetime of neodymium ions is presented in Fig. 7.

The pump pulses were coupled by a prism coupler to the slab waveguide. A mechanical chopper was used to modulate the pump beam (in a focal plane) with a 15 μs falltime and 10 ms repetition period. A monochromator operating at 1060nm with low resolution (40nm bandpath) was used to isolate the particular transition from $^4F_{3/2}$ to $^4I_{11/2}$. The signal generated by the photodiode was analysed with a digital oscilloscope.

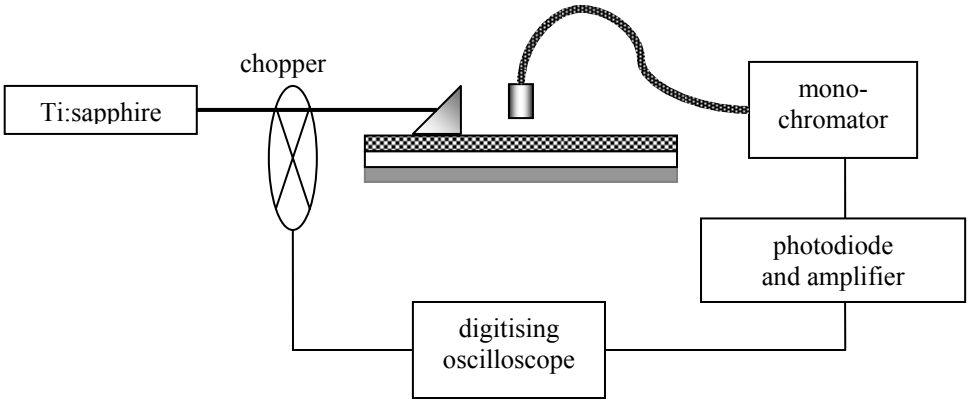


Fig. 7 Experimental setup for luminescence lifetime measurement.

4.2 Luminescence lifetime

Fig. 8 gives the time dependence decay of the luminescent intensity recorded by the oscilloscope. The luminescence lifetime is 130μs obtained by an exponential fit. This value is of the same order of magnitude as the typical value (around 200 μs) and the value from our Judd-Ofelt analysis (182 μs) , which indicates that the transition properties of the neodymium ions within our polymer host is good.

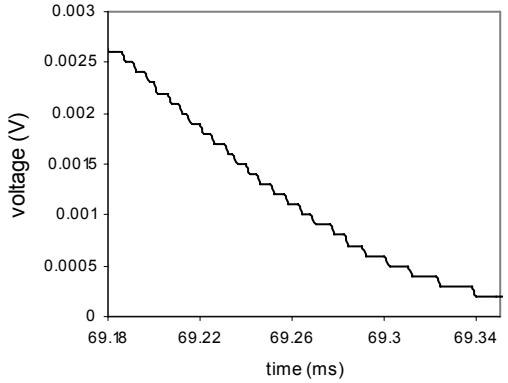


Fig. 8 Decay curve of $Nd(TTA)_3$phen doped 6-FDA/epoxy.

5. SMALL SIGNAL GAIN

5.1 Rate equations

With the aid of the rate equations and the parameters obtained from our experiments, we estimated the optical gain for neodymium ions. Theoretically neodymium can be described as a four level system as shown in Fig 9. The neodymium ions are excited from the ground state $^4I_{9/2}$ to the upper state $^4F_{5/2}$ followed by a fast decay to the excited state $^4F_{3/2}$.

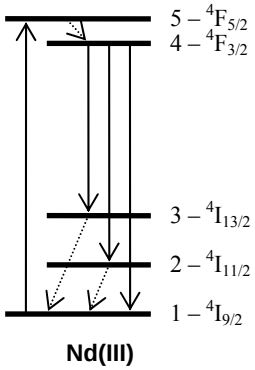


Fig. 9 Energy diagram of Nd^{3+} ions.

The rate equations can be simplified as follows [16, 17]:

$$\frac{dN_1}{dt} = -W_{15}N_1 + W_{42}N_4 + \frac{N_4}{\tau}$$
$$\frac{dN_4}{dt} = W_{15}N_1 - W_{42}N_4 - \frac{N_4}{\tau} = -\frac{dN_1}{dt} \qquad (3)$$
$$N_1 + N_4 = N_{tot}$$

At steady state, we can solve the above equations and get the results:

$$N_1 = \left(\frac{W_{42} + \frac{1}{\tau}}{W_{42} + \frac{1}{\tau} + W_{15}} \right) N_{tot} \qquad N_4 = \left(\frac{W_{15}}{W_{42} + \frac{1}{\tau} + W_{15}} \right) N_{tot} \qquad (4)$$

where τ is the lifetime of neodymium ions. W_{15} and W_{42} are the pump absorption and signal emission rates, which are defined as follows:

$$W_{15} = \frac{\sigma_a}{h\nu_p}\frac{P_p}{A} = \sigma_a \Phi_p \qquad W_{42} = \frac{\sigma_e}{h\nu_p}\frac{P_s}{A} = \sigma_e \Phi_s \tag{5}$$

A is the cross section of the waveguide. σ_a and σ_e are the absorption and emission cross section of neodymium ions. P_p and P_s are the powers of pump and signal. The change in photon flux and thus the power change of the signal and pump along the waveguide are given by:

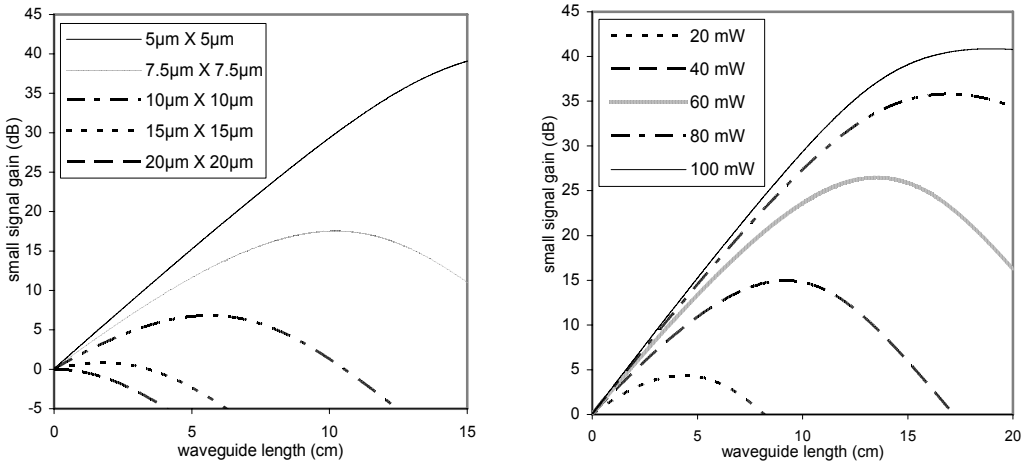
$$d\Phi_s = W_{42} N_4 dz \qquad d\Phi_p = W_{15} N_1 dz \tag{6}$$

5.2 Optical gain

The optical gain properties of Nd doped channel waveguides can be predicted based on the method described above. Results are presented in Fig.10 for varying waveguide cross section and pump power using the parameters listed in Table 3.

Table 3 Simulation parameters for the Nd-complex doped channel waveguides; λ_p and λ_s are the wavelengths of pump and signal; N is the concentration of Nd^{3+} in the waveguides.

Parameter	Value
λ_p	800 *nm*
λ_s	1060 *nm*
P_p	100 *mW*
P_s	0.001 *mW*
σ_a	$5.92\times10^{-24} m^2$
σ_e	$7.54\times10^{-24} m^2$
τ	0.13 *ms*
N	$1.1\times10^{25} m^{-3}$



(a) (b)

Fig. 10 Small signal gain for Nd doped waveguides as a function of waveguide length; (a) for various cross sections when pumped at 100mW; (b) for a fixed cross section of 5μm X 5μm for various pump powers.

The optical gain of multimode waveguides drops rapidly with increasing waveguide cross section. High pump power and waveguides with small cross sections are needed for gain measurement. A waveguide with a dimension of $5\mu m \times 5\mu m$ is expected to provide gain of more than 2dB/cm at pump power of 40mW.

6. CONCLUSION

Nd-complex doped slab as well as channel waveguides were realized. The emission around 890, 1060 and 1330nm indicates that the Nd ions in the polymer waveguides are active. The pump power dependent emission spectra for Nd-complex doped channel waveguide have been studied and results are quite promising for optical amplification. The luminescence lifetime from measurement shows a good agreement with the values from literature and Judd-Ofelt theory. It demonstrates that the neodymium ions within our polymer host have good transition properties. The nonlinear response observed in the emission spectrum at high pump powers is an indication of stimulated emission in this Nd complex doped sample. With a sufficient pump power and suitable cross section of the waveguide, gain can be achieved. Based on the experimental study and small signal gain model, we arrive at the conclusion that the $Nd(TTA)_3$phen-doped 6-FDA/epoxy planar waveguides are well suited as gain media for optical amplification.

7. ACKNOWLEDGEMENT

The authors acknowledge the financial support of STW (project 06986).

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