Winter College on Fibre Optics, Fibre Lasers and Sensors

12 - 23 February 2007

Spectroscopy of Rare Earth Doped Glasses

(part 2)

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Spectroscopy of Rare Earth Doped Glasses

Lecture II

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Lessons Plan

Part II – Upconversion spectroscopy and Applications of REDG

II.1 Up-conversion Spectroscopy
II.2 REDG Ceramics
   Applications of REDG
II.3 REDG for Lasers
II.4 REDG for Fiber Lasers and Amplifiers
II.5 REDG Planar and Channel Waveguides
II.6 REDG Microbarcodes

Literature
Upconversion spectroscopy


Upconversion and Anti-Stokes Processes with f and d Ions in Solids

François Auzel

GOTR, UMR 7574-CNRS, 1, Place A-Briand, 92195 Meudon Cedex, France
Figure 1. Various basic energy transfer processes between two ions considered before 1966: note that activator ion (A) receiving the energy from the sensitizer (S) is initially in its ground state. Cross-relaxation is the special case where S is identical to A. Doubled arrows symbolize the Coulombic interaction: (a) radiative resonant transfer; (b) resonant nonradiative transfer; (c) phonon-assisted nonradiative transfer; (d) cross-relaxation special case of nonradiative transfer.
Probability for such transfer between two ions at a sufficiently large distance $R$ is found to be\cite{20}

$$p_{SA}(R) = \frac{\sigma_a}{4\pi R^2 \tau_s} \int g_s(\nu)g_a(\nu) \, d\nu$$  \hspace{1cm} (1)

$R^{-2}$ dependence allows long range energy diffusion \hspace{1cm} \rightarrow \hspace{1cm} \text{photon trapping effects}

Photon trapping increases apparent experimental lifetime!
Let us take as example case 1(b)

For dipole-dipole interaction, the transfer probability can be written as (Förster, 1948):

\[ p_{SA} = \frac{1}{\tau_S} \left( \frac{R_0}{R} \right)^6 \]

(Dexter, 1953)

The energy transfer probability for electric multipolar interactions can be more generally written as:

\[ p_{SA} = \frac{(R_0/R)^s}{\tau_S} \]  (4)

where \( s \) is a positive integer taking the following values:

- \( s = 6 \) for dipole–dipole interactions,
- \( s = 8 \) for dipole–quadrupole interactions,
- \( s = 10 \) for quadrupole–quadrupole interactions.
Mechanisms for upconversion

Single ion resonant processes

(a) Sequential TPA (Two photon absorption) (or more!)
(b) SHG (second harmonic generation)
(c) TPA

\[ |1\rangle \quad |2\rangle \quad |x\rangle \]

(a) (b) (c)
Two ions resonant processes

**Figure 2.** APTE basic step: energy transfer toward an ion already in an excited state. Nonradiative energy transfer is either resonant or phonon-assisted with energy mismatch $\epsilon_0 \neq 0$. 
Two photon upconversion processes efficiencies

**Figure 3.** Various two-photon upconversion processes with their relative efficiency in considered materials.
Figure 5. Cooperative (a) and APTE (b) energy scheme for $n$-photon ($n = 1-5$) upconversion in Er$^{3+}$-doped hosts.
Figure 20. First operating APTE upconversion pulsed laser-pumping schemes in Yb–Ho and Yb–Er couples. (Reprinted with permission from ref 218. Copyright 1971 American Institute of Physics.)
IR pumped upconversion in thulium doped fiber
Cross relaxation

\[ E_i - E_f \approx E_1 \]

- \[ |i\rangle \]
- \[ |f\rangle \]
- \[ |1\rangle \]

- Ion A
- Ion B

- Leads to FLUORESCENCE QUENCHING

- Strong dependence on ions concentration
Fig. 6. Lifetime values of $^3P_0$ and $^1D_2$ levels as a function of concentration at 4.2 K. Lifetimes were obtained by exciting at 486 nm and collecting the luminescence at 525 and 606 nm, respectively.

5. Conclusions

From the above results, the following conclusions can be reached:

1. Fluorescence quenching from the $^1D_2$ state has been demonstrated to occur for Pr$^{3+}$ concentrations higher than 0.1 mol% even at 4.2 K. This can be attributed to a cross relaxation process.

2. The time evolution of the decays from the $^1D_2$ state for concentrations higher than 0.1 mol% is consistent with a dipole-dipole energy transfer mechanism.

R. Balda, Fernández, I. Saéz de Ocáriz, J. L. Adam, A. Mendioroz and E. Montoya
1.5 μm Emission and infrared-to-visible frequency upconversion in Er$^{3+}$/Yb$^{3+}$-doped phosphoniobate glasses

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Available online 24 July 2006

Abstract

Sodium phosphoniobate glasses with the composition (mol\%$)$ 75NaPO$_3$–25Nb$_2$O$_5$ and containing 2 mol\% Yb$^{3+}$ and $x$ mol\% Er$^{3+}$ ($0.01 \leq x \leq 2$) were prepared using the conventional melting/casting process. Er$^{3+}$ emission at 1.5 μm and infrared-to-visible upconversion emission, upon excitation at 976 nm, are evaluated as a function of the Er$^{3+}$ concentration. For the lowest Er$^{3+}$ content, 1.5 μm emission quantum efficiency was 90%. Increasing the Er$^{3+}$ concentration up to 2 mol\%, the emission quantum efficiency was observed to decrease to 37% due to concentration quenching. The green and red upconversion emission intensity ratio was studied as a function of Yb$^{3+}$ co-doping and the Er$^{3+}$–Er$^{3+}$ energy transfer processes.

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II.1 Upconversion Spectroscopy

Fig. 1. Room temperature absorption spectra (a) 2.0 mol% Er$^{3+}$ and (b) 2.0 mol% Yb$^{3+}$ and 2.0 mol% Er$^{3+}$.

Table 1  
Experimental ($P_{\text{EXP}}$) and calculated ($P_{\text{ED}}$) oscillator strengths obtained for the 2 mol% Er$^{3+}$ glass sample, and Judd-Ofelt intensity parameters $\Omega_2 = 7.5 \times 10^{-20}$ cm$^2$, $\Omega_4 = 1.4 \times 10^{-20}$ cm$^2$ and $\Omega_6 = 0.7 \times 10^{-20}$ cm$^2$

<table>
<thead>
<tr>
<th>Er$^{3+}$ transition $^4I_{15/2} \rightarrow \tilde{v}$ (cm$^{-1}$)</th>
<th>$P_{\text{EXP}}$ (10$^{-6}$)</th>
<th>$P_{\text{ED}}$ (10$^{-6}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^4G_{11/2}$</td>
<td>26410</td>
<td>21.2</td>
</tr>
<tr>
<td>$^2H_{9/2}$</td>
<td>24570</td>
<td>0.49</td>
</tr>
<tr>
<td>$^4F_{5/2} + ^4F_{3/2}$</td>
<td>22173</td>
<td>0.41</td>
</tr>
<tr>
<td>$^4F_{7/2}$</td>
<td>20490</td>
<td>1.37</td>
</tr>
<tr>
<td>$^2H_{11/2}$</td>
<td>19138</td>
<td>11.8</td>
</tr>
<tr>
<td>$^4S_{3/2}$</td>
<td>18381</td>
<td>0.28</td>
</tr>
<tr>
<td>$^4F_{9/2}$</td>
<td>15310</td>
<td>1.72</td>
</tr>
<tr>
<td>$^4I_{9/2}$</td>
<td>12499</td>
<td>0.29</td>
</tr>
<tr>
<td>$^4I_{11/2}$</td>
<td>10235</td>
<td>0.52</td>
</tr>
<tr>
<td>$^4I_{13/2}$</td>
<td>6553</td>
<td>1.03 (P$_{\text{MD}}$ excluded)</td>
</tr>
</tbody>
</table>

The RMS of the fitting procedure was $9.3 \times 10^{-8}$. 


II.1 Upconversion Spectroscopy

Fig. 2. Room temperature infrared emission spectrum for the 2.0 mol\% Er$^{3+}$ sample under 976 nm excitation.

Fig. 3. Er$^{3+}$ and Yb$^{3+}$ free ion energy levels. The arrows indicate the excitation and relaxation processes discussed in the text.
II.1 Upconversion Spectroscopy

Fig. 5. Er\textsuperscript{3+} \textsuperscript{4}I\textsubscript{13/2} level lifetime values as a function of Er\textsuperscript{3+} concentration. The line is just a guide for the reader.

Fig. 6. Upconversion emission spectra as a function of the excitation power at 976 nm: (a) 2 mol\% Yb\textsuperscript{3+} and 1.0 mol\% Er\textsuperscript{3+} sample, (b) 2 mol\% Yb\textsuperscript{3+} and 2.0 mol\% Er\textsuperscript{3+} sample. The 976 nm pump power increase is indicated by the arrow for values of 100, 120, 150, 200, and 230 mW.
Blue upconversion enhancement by a factor of 200 in Tm$^{3+}$-doped tellurite glass by codoping with Nd$^{3+}$ ions

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FIG. 3. Energy level schematic and the relevant channels responsible for the blue upconversion at 480 nm.

FIG. 1. Upconverted signal for (a) tellurite glass doped with Tm$^{3+}$ (0.2 mol %); (b) ZBLAN glass doped with Tm$^{3+}$ (0.2 mol %); (c) tellurite glass codoped with Tm$^{3+}$ (0.2 mol %) and Nd$^{3+}$ (0.5 mol %); (d) ZBLAN glass codoped with Tm$^{3+}$ (0.2 mol %) and Nd$^{3+}$ (0.5 mol %), upon near-infrared (795 nm) excitation.
Cooperative absorption

\[ E_{\text{photon}} = \hbar \omega = \hbar \omega_a + \hbar \omega_b \]

\[ E_a = \hbar \omega_a \]

\[ E_b = \hbar \omega_b \]

See more in Auzel’s review article
Photon avalanche


Figure 23. Decrease of transmission in a Pr\textsuperscript{3+}:LaCl\textsubscript{3} sample under \textsuperscript{3}H\textsubscript{5}–\textsuperscript{3}P\textsubscript{1} pumping. (Reprinted with permission from ref 12. Copyright 1990 Elsevier.)
Diode pumped avalanche upconversion in Pr$^{3+}$-doped fibers

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REDG Ceramics

Several products:

Cook-top panels
dinnerware
electronics
medicine
dentistry

• Tough materials
• Zero ou negative thermal expansion
• Can be made TRANSPARENT!

http://www.ch.seikei.ac.jp/kojima/Environmental/index%201.htm
Rare-earth-doped transparent glass ceramics

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Received 15 April 2002; accepted 16 October 2002

Abstract — Glass ceramics are a known class of polycrystalline ceramic materials, where, depending on the glass matrix and the particular crystalline phases, one can obtain materials with improved mechanical, thermal, electrical or optical properties. The characteristics and applications of optical glass ceramics are reviewed, with particular emphasis on rare-earth-doped transparent glass ceramics for photonics, including the search for new transparent glass ceramic compositions and the development of suitable methods to process such materials into functional devices. To cite this article: M. Clara Gonçalves et al., C. R. Chimie 5 (2002) 845–854 © 2002 Académie des sciences / Éditions scientifiques et médicales Elsevier SAS

rare earth / glass ceramics / photonics

Rare Earth doped transparent glass-ceramics
M. Mortier, M. Génotelle, G. Patriarche

http://www.solgel.com/articles/Dec00/glass/envitromm.html
REDG Ceramics

• Crystal sizes well below incident light wavelength present negligible attenuation due to scattering! (Rayleigh-Gans theory).

• Requires a refractive index difference <0.1 between amorphous and crystalline phases.

Driving applications:

- large telescope mirror blanks
- liquid crystal displays
- solar cells
- photonic devices (lasers, amplifiers, upconverters, etc)
Rare Earth doped transparent glass-ceramics
M. Mortier, M. Génotelle, G. Patriarche

Germanate oxyfluorides glass of the family:
\[(50\text{GeO}_250-\text{yPbO}\text{yPbF}_2+x\text{ErF}_3)\]
\[y, \ y=\{10,20\} \ x=\{0,4\}\]
Rare Earth doped transparent glass-ceramics
M. Mortier, M. Génotelle, G. Patriarche
Other glass ceramics:

Silicate oxyfluoride
Tellurite oxyhollides
and more (see M C G review).
Applications of REDG

II.3 REDG for Lasers
II.4 REDG for Fiber Lasers and Amplifiers
II.5 REDG Planar and Channel Waveguides
II.6 REDG Microbarcodes
REDG for Lasers

Nd:YAG (crystal), Nd:Glass

II.3 REDG for LASERS
Typical pump geometries

- **End pumped laser rod**
  - focusing optic
  - laser rod
  - laser beam
  - diode laser
  - HR-mirror
  - out coupling mirror

- **Side pumped laser rod**
  - laser rod
  - focusing optic
  - laser beam
  - HR-mirror
  - out coupling mirror
  - diode laser
Niche application

Electronic Control System for Table-Top Terawatt Nd:Glass Laser
REDG for Fiber Lasers and Amplifiers

Fiber lasers

- pump light
- mirror
- fiber jacket
- active core
- mirror
- laser output
REDG for Fiber Lasers and Amplifiers

Laser transitions in rare-earth-doped fluoride glass fibers
II.3 REDG for Fiber Lasers and amplifiers

Fiber Laser: emission wavelength

first demonstration of a fiber laser: in the early sixties!

II.3 REDG for Fiber Lasers and amplifiers

Er: upconversion fiber laser
II.3 REDG for Fiber Lasers and amplifiers

Thulium doped upconversion fiber laser
Fiber Amplifiers

II.3 REDG for Fiber Lasers and amplifiers

Rare-earth-doped glasses for fiber amplifiers in broadband telecommunication

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Received 30 April 2002; accepted 28 May 2002

Material Design for Rare Earth Laser Host

Local Structure

Mössbauer spectra

Absorption Cross Section

Judd-Olfelt parameters

4-level

3-level

Fluorescence Lifetime

Emission Cross Section

Vibrational property

Phonon Sideband

Radiative Quantum Efficiency

Energy Transfer Efficiency

Compositional Design of Excellent Host

Fig. 2. Research scheme for efficient laser materials.
Optical Amplifiers Diversity

REDFA, such as:

**PDFA**

- $^1G_4$
- $^3F_4$
- $^3H_5$
- $^3H_4$

**EDFA**

- $^4I_{11/2}$
- $^4I_{13/2}$
- $^4I_{15/2}$

**TDFA**

- $^3F_{2&3}$
- $^3H_4$
- $^3F_4$
- $^3H_5$
- $^3H_6$

**HYBRIDS**

- **parallel**
- **series**
Importance of the host glass

II.3 REDG for Fiber Lasers and amplifiers

Fig. 3. Fluorescence spectra of Er$^{3+}$-doped glasses: (a) Bi-silicate, (b) Bi-borate, (c) Tellurite and (d) Al-silica.
Importance of understanding REE S-band (1450nm-1510nm) TDFA

+ S-band emission: $^3\text{H}_4 \rightarrow ^3\text{F}_4$

+ Conversion efficiency
+ Low loss in non-operation
+ Diode pump sources

- Multi-phonon relaxation
- Material reliability
- Lifetime bottleneck
- Complex pump schemes
TDFA – Pumping Schemes

**Single wavelength pump**

Gain and NF as function of the signal wavelength. The pump powers are:
- 400mW, ■
- 300mW and ●
- 150mW, ▲

Dual-wavelength pump schemes (preferred, more efficient)

References

Novel dual wavelength (1050 nm + 800 nm) pumping scheme for thulium doped fiber amplifiers
A.S.L. Gomes, M.L. Sundheimer, M.T. Carvalho, J.F. Martins-Filho, C.J.A. Bastos-Filho, Univ. Federal de Pernambuco, Brazil; W. Margulis, ACREO, Sweden. Contact e-mail: anderson@df.ufpe.br
Results for the 800+1050nm pumping scheme

Single pumped:
1050nm (80mW)

Dual pumped:
1050nm (80mW) +800nm (73mW)

Results for the 800+1050nm pumping scheme

$L = 18 \text{m}, 2000 \text{ppm ZBLAN, } P_s = -27 \text{dBm}$
II.3 REDG for Fiber Lasers and Amplifiers

Fiber Amplifiers

Chirped-pulse-amplification

short pulse

stretched pulse

amplified short pulse

stretcher

amplifier

amplified pulse

compressor

II.3 REDG for Fiber Lasers and amplifiers

Fiber Amplifiers

CPA-fiber amplifier – power amplifier

diode laser
976 nm

Yb-doped large-mode-area fiber
length: 13.5 m
core: $\varnothing = 28.5 \, \mu m$, NA = 0.06, MFD = 23 $\mu$m
inner clad.: $\varnothing = 400 \, \mu m$, NA = 0.38, D
doping: 700 ppm (mol) Yb$_2$O$_3$

beam quality:
diffraction-limited ($M^2 = 1.1$)
degree of polarization:
~ 50% (3 to 1)

output characteristics

Slope efficiency ~80%

Launched pump power [W]

Output power [W]
Fiber Amplifiers

CPA-fiber amplifier – power amplifier

diode laser 976 nm

Yb-doped large-mode-area fiber
core: $\varnothing = 28.5 \, \mu m$, NA = 0.06
doping: 700 ppm (mol) Yb$_2$O$_3$
inner clad.: $\varnothing = 400 \, \mu m$, NA = 0.38, D
length: 13.5 m

emitted spectrum at 140 W

pulse duration at 140 W
Fiber Amplifiers

CPA-fiber amplifier – compressor stage

transmission gratings in fused silica fabricated by electron beam lithography
grating separation: 61 cm diffraction angle: 44.5° (3° off Littrow)

Output

compressor efficiency

autocorrelation trace

80 W average power of 400 fs pulses

II.3 REDG for Fiber Lasers and amplifiers
REDG Planar and Channel Waveguides

II.4 REDG planar and channel waveguides

Planar Waveguide Fabrication Facilities
Departamento de Física - UFPE

Clean Room (class 1000)
(mask alignment, photolithography, etc)

Preparation methods employed:
- Proton exchange and Ti-indiffusion for LiNbO₃
- Ion Exchange for silicate glasses
- Nonmetallic indiffusion for fluoroindate glasses

Characterization & Applications
LINEAR (losses, modes, Δn, etc)
NONLINEAR (e-o modulators, all-optical switches, lasers, amplifiers, etc)

End Fire Coupling Into Waveguides
All-optical switching in rare-earth doped channel waveguide

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(Received 26 August 1994; accepted for publication 11 November 1994)

The operation of an all-optical switch in a rare-earth doped channel waveguide is described. The switching mechanism is based on an optically induced intramodal energy exchange, driven by a resonantly enhanced nonlinearity of a Nd$^{3+}$ ion. Switching times around 410 $\mu$s at a repetition rate of 1 kHz was demonstrated. © 1995 American Institute of Physics.


Ion exchange, 8mm long, 2-10$\mu$m width, $\Delta n = 8.7 \times 10^{-3}$
Silicate glass, 16%Na$_2$O, 2%Nd$_2$O$_3$, K$^+$ ↔ Na$^+$
II.4 REDG planar and channel waveguides

PHYSICAL MECHANISM FOR ALL-OPTICAL SWITCHING IN Nd-DOPED WAVEGUIDE

Energy exchange between two-lobes of a high order propagating mode.

First observed in RED optical fibres (Pantell et al. OL 92; Sadowski et al. OL 93). Fibre lengths of ~1m were employed.

The pump beam resonantly induces a differential phase shift in the two spatial components of the signal beam.

As a consequence, a change in intensity occurs from one lobe to the other.

MAIN FEATURES:

The nature of the optical nonlinearity is electronic in origin.

Repetition rate limited by pump level lifetime (410μs).

Pump Power dependence: Linear

Estimated $n_2 \sim 10^{-13} \text{cm}^2/\text{W}$
Fabrication and Characterization of Photonic Devices Directly Written in Glass Using Femtosecond Laser Pulses

Catalin Florea, Member, IEEE, Member, OSA, and Kim A. Winick, Senior Member, IEEE, Member, OSA

- writing geometry:

- pulse energy: 0.1-10 μJ
- laser wavelength: 800 nm
- pulse repetition rate

longitudinal writing

transverse writing
C-band waveguide amplifier produced by femtosecond laser writing

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8 August 2005 / Vol. 13, No. 16 / OPTICS EXPRESS 5976

Fig. 1. Optical waveguide amplifier configuration and characterization set-up.

Fig. 2. Measured internal gain spectrum obtained with an incident pump power of 460 mW in bi-directional pumping configuration. The dashed line indicates the total insertion losses.
Rare earth-doped glass microbarcodes

Matthew J. Dejneka*, Alexander Streitsov, Santona Pal, Anthony G. Frutos, Christy L. Powell, Kevin Yost, Po Ki Yuen, Uwe Müller, and Joydeep Lahiri*

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PNAS 2003;100;389-393; originally published online Jan 6, 2003;
doi:10.1073/pnas.0236044100

• Employs μm size glass barcodes
• UV excited fluorescences
• APPLICATION: Bioessays

Advantages of REDG

- High quantum efficiencies
- Noninterference with common fluorescence labels
- Inertness to most organics and aqueous solvents

$> 10^6$ distinguishable possibilities
Rare earth-doped glass microbarcodes

Matthew J. Dejneka*, Alexander Streltsov, Santona Pal, Anthony G. Frutos, Christy L. Powell, Kevin Yost, Po K. Yuen, Uwe Müller, and Joydeep Lahiri*

Fabrication of Barcodes. Conventional optical fiber draw methods were used to fabricate the encoded fiber ribbons. First, the optimized glasses were melted and cast into 25 × 25 mm square bars and annealed for 1 h at 750°C. These bars were drawn into lengths of square (3.5-mm sides) canes and stacked in a pre-determined order to define a barcode pattern. The assembly was then fused in a graphite press in a furnace at 900°C under N2. The fused preform was drawn at 1,200°C into a ribbon fiber (20 μm thick, 100 μm wide). The ribbon fiber was scribed every 20 μm at a rate of 5 mm/s with 800-nm femtosecond laser pulses (100 mW average power) by using a computer-controlled stage. The scribed ribbon fiber was then sonicated for 60 s in water to break the ribbon along the scribes into individual barcodes.
II.5 REDG microbarcodes

Fig. 3. False-color image of two 100 × 20 μm barcodes (met) and corresponding fluorescence spectrum barcode elements. The same color scheme is used for the spectra and the image (e.g., the yellow band in the barcode corresponds to the yellow (combination Tm + Dy) line spectrum).

Fig. 4. Fluorescence false-color images of barcode particles A and B used in a DNA hybridization assay using Cy3-labeled DNA. (a) “White Light” image. (b) Cy3 channel image. (c) Rl images obtained by using a 420 nm long-pass filter.
Break time!!!!!!