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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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The Abdus Salam International Centre for Theoretical Physics



International Atomic Energy Agency

Winter College on Fibre Optics, Fibre Lasers and Sensors (12 - 23 February 2007)

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Spectroscopy of Rare Earth Doped Glasses

Lessons Plan

Part II – Upconversion spectroscopy and Applications of REDG

II.1 Up-conversion Spectroscopy
II.2 REDG Ceramics

<u>Applications of REDG</u>

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

Chem. Rev. 2004, 104, 139-173

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

François Auzel

GOTR, UMR 7574-CNRS, 1, Place A-Briand, 92195 Meudon Cedex, France



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Energy Transfer



Chemical Reviews, 2004, Vol. 104, No. 1 141

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 be²⁰

$$p_{\rm SA}(R) = \frac{\sigma_{\rm A}}{4\pi R^2 \tau_{\rm S}} \int g_{\rm S}(\nu) g_{\rm A}(\nu) \,\mathrm{d}\nu \tag{1}$$

R⁻² dependence allows long range energy diffusion → 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) :

$$p_{\rm SA} = \frac{1}{\tau_{\rm S}} \left(\frac{R_0}{R}\right)^6$$

(Dexter, 1953)

The energy transfer probability for electric multipolar interactions can be more generally written as²⁷

$$p_{\rm SA} = \frac{\left(R_0/R\right)^s}{\tau_{\rm S}} \tag{4}$$

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





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 Er³⁺-doped hosts.

Chemical Reviews, 2004, Vol. 104, No. 1 159



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.)

Spectroscopy





IR pumped upconversion in thulium doped fiber







Cross relaxation



• Leads to FLUORESCENCE QUENCHING

•Strong dependence on ions concentration

II.1 Upconversion Spectroscopy



Fig. 6. Lifetime values of ${}^{3}P_{0}$ and ${}^{1}D_{2}$ 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.



Fig. 2. Energy levels diagram of Pr^{3+} in NBGd fluorophosphate glass (from the absorption data).

5. Conclusions

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

- Fluorescence quenching from the ¹D₂ state has been demonstrated to occur for Pr³⁺ 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 ${}^{1}D_{2}$ state for concentrations higher than 0.1 mol% is consistent with a dipole–dipole energy transfer mechanism.

R. Balda, Fernándeza, I. Saéz de Ocáriza, J. L. Adam, A. Mendioroz and E. Montoya Opt. Mat. 13, 159-165 (1999)





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Journal of Non-Crystalline Solids 352 (2006) 3636-3641

JOURNAL OF NON-CRYSTALLINE SOLIDS

www.elsevier.com/locate/jnoncrysol

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%) 75NaPO₃-25Nb₂O₅ and containing 2 mol% Yb³⁺ and x mol% Er³⁺ (0.01 $\leq x \leq 2$) were prepared using the conventional melting/casting process. Er³⁺ emission at 1.5 µm and infrared-to-visible upconversion emission, upon excitation at 976 nm, are evaluated as a function of the Er³⁺ concentration. For the lowest Er³⁺ content, 1.5 µm emission quantum efficiency was 90%. Increasing the Er³⁺ 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 Yb³⁺ co-doping and the Er³⁺ energy transfer processes.





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 ($P_{\rm EXP}$) and calculated ($P_{\rm ED}$) oscillator strengths obtained for the 2 mol% Er³⁺ glass sample, and Judd–Ofelt intensity parameters $\Omega_2 = 7.5 \times 10^{-20}$ cm², $\Omega_4 = 1.4 \times 10^{-20}$ cm² and $\Omega_6 = 0.7 \times 10^{-20}$ cm²

${\rm Er}^{3+} \ transition \ ^4I_{15/2} {\rightarrow}$	$\bar{v} (cm^{-1})$	$P_{\rm EXP} (10^6)$	$P_{\rm ED}~(10^6)$
⁴ G _{11/2}	26410	21.2	21.1
² H _{9/2}	24570	0.49	0.47
${}^{4}\text{F}_{5/2} + {}^{4}\text{F}_{3/2}$	22173	0.41	0.57
⁴ F _{7/2}	20490	1.37	1.38
$^{2}H_{11/2}$	19158	11.8	11.9
⁴ S _{3/2}	18381	0.28	0.30
⁴ F _{9/2}	15310	1.72	1.72
⁴ I _{9/2}	12499	0.29	0.33
⁴ I _{11/2}	10235	0.52	0.52
⁴ I _{13/2}	6553	$1.03 (P_{MD} \text{ excluded})$	0.90

The RMS of the fitting procedure was 9.3×10^{-8} .





Fig. 2. Room temperature infrared emission spectrum for the 2.0 mol% ${\rm Er}^{3+}$ 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.





Fig. 5. $Er^{3+4}I_{13/2}$ level lifetime values as a function of Er^{3+} concen 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^{3+} and 1.0 mol% Er^{3+} sample, (b) 2 mol% Yb^{3+} and 2.0 mol% Er^{3+} sample. The 976 nm pump power increase is indicated by the arrow for values of 100, 120, 150, 200, and 230 mW.



COMMUNICATIONS

Blue upconversion enhancement by a factor of 200 in Tm³⁺-doped tellurite glass by codoping with Nd³⁺ ions

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Y. Messaddeq, F. C. Cassanjes, G. Poirier, and S. J. L. Ribeiro Instituto de Química, Universidade do Estado de São Paulo, P.O. Box 355, 14801-970 Araraquara-SP, Brazil



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 ${\rm Tm}^{3+}$ (0.2 mol %); (b) ZBLAN glass doped with ${\rm Tm}^{3+}$ (0.2 mol %); (c) tellurite glass codoped with ${\rm Tm}^{3+}$ (0.2 mol %) and ${\rm Nd}^{3+}$ (0.5 mol %); (d) ZBLAN glass codoped with ${\rm Tm}^{3+}$ (0.2 mol %) and ${\rm Nd}^{3+}$ (0.5 mol %); upon near-infrared (795 nm) excitation.



Cooperative absorption



See more in Auzel's review article



Photon avalanche

Case, W. E.; Koch, M. E.; Kueny, A. W. J. Lumin. 1990, 45, 351.



Figure 23. Decrease of transmission in a Pr^{3+} :LaCl₃ sample under ${}^{3}H_{5}-{}^{3}P_{1}$ pumping. (Reprinted with permission from ref 12. Copyright 1990 Elsevier.)



UPCONVERTED EMISSION (a.u.)

1.0

0.8

0.6

0.4

0.2

0.0 400

 $^{\prime}S_{0} \rightarrow ^{\prime}I_{6}$

450

500

WAVELENCTH (nm)

Optics Communications Volume 103, Issues 5-6 , 1 December 1993, Pages 361-364

Diode pumped avalanche upconversion in Pr³⁺-doped fibers

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Universidade Federal de Pernambuco, Departamento de Física, 50739, Recife, Brazil

50

22

10

(10[°]cm⁻¹)

ENERGY



 ${}^{3}P_{\theta} \rightarrow {}^{3}H_{A}$

 ${}^{3}P_{0} \rightarrow {}^{3}H_{6}$

550











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



REDG Ceramics

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 : $(50GeO_250-yPbO_yPbF_2+xErF_3)$ y, y=[10,20] x=[0,4]

II.2 REDG Ceramics



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Rare Earth doped transparent glass-ceramics M. Mortier, M. Génotelle, G. Patriarche M. Clara Gonçalves et al. / C. R. Chimie 5 (2002) 845-854



Fig. 2. Normalised photoluminescence spectra of Er^{3+} (${}^{4}\mathrm{I}_{13/2} \rightarrow {}^{4}\mathrm{I}_{15/2}$ transition) in the precursor oxyfluoride glass (thick solid line) and in a developed glass ceramic heat treated at 440 °C for 5 h (dashed line). (Adapted from ref. [38].)

Other glass ceramics:

Silicate oxyfluoride Tellurite oxyhallides and more (see M C G review).

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



Nd

REDG for Lasers

Nd:YAG (crystal), Nd:Glass

Yb

4**H**_{9/2} Energy [x 1000 cm⁻¹] 12 10 10 cm⁻¹ Energy [x 1000 cm⁻¹ 12 ${}^{4}\mathbf{F}_{3/2}$ ${}^{2}\mathbf{F}_{5/2} \mathbf{f}$ 1350 nm 10 808 nm 920 nm 1060 nr 8 1035 nm 1006 nm 1052 nm 1035 nm 1140 nm 087 nm 909 nm 860 nm 962 nm 907 nm 975 nm 909 nm 962 nm 975 nm б ${}^{4}I_{15/2}$ 4 ${}^{4}I_{13/2}$ ${}^{4}\mathbf{I}_{\mathbf{11/2}}$ 2 2 ${}^{2}\mathbf{F}_{7}$ 0 0 ${}^{4}\mathbf{I}_{9/2}$ Fraunhofer Institut Institute of Applied Physics Angewandte Optik und Feinmechanik



Typical pump geometries

end pumped laser rod





Fraunhofer Institut Angewandte Optik und Feinmechanik



Institute of Applied Physics



Niche application

Electronic Control System for Table-Top Terawatt Nd:Glass Laser





REDG for Fiber Lasers and Amplifiers







Fiber Laser: emission wavelength



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

E. Snitzer, "Neodymium glass laser," Proc. of the Third International conference on Solid Lasers, Paris, page 999 (1963). C.J. Koester and E.Snitzer, "Amplification in a fiber laser," Appl. Opt. 3, 10, 1182 (1964).





Er: upconversion fiber laser







Thulium doped upconversion fiber laser





Fiber Amplifiers

 C. R. Chimie 5 (2002) 815–824
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Rare-earth-doped glasses for fiber amplifiers in broadband telecommunication

Setsuhisa Tanabe*

Faculty of Integrated Studies, Kyoto University, Kyoto 606-8501, Japan Received 30 April 2002; accepted 28 May 2002

Material Design for Rare Earth Laser Host

Local Structure Mbssbauer spectra



Fig. 2. Research scheme for efficient laser materials.



Optical Amplifiers Diversity

REDFA, such as:





Importance of the host glass



Fig. 3. Fluorescence spectra of $Er^{3+}\mbox{-doped glasses;}$ (a) Bi-silicate, (b) Bi-borate, (c) Tellurite and (d) Al-silica.





Importance of understanding REE S-band (1450nm-1510nm) TDFA



+ S-band emission: ${}^{3}H_{4} \rightarrow {}^{3}F_{4}$

- + Conversion efficiency
- + Low loss in non-operation
- + Diode pump sources
- Multi-phonon relaxation
- Material reliability
- Lifetime bottleneck
- Complex pump schemes

TDFA – Pumping Schemes

II.3 REDG for Fiber Lasers and amplifiers

Single wavelength pump ¹G₄ 20 16 12 ³F₂ ³F₃ Gain [dB] 10 ³H₄ 12 1,05µm ${}^{3}H_{5}$ 8 0 2,3µm NF [dB] $^{3}F_{4}$ 1,05µm 1,47µm 1455 1460 1465 1470 1475 1480 1485 1490 Wavelength [nm] ${}^{3}H_{6}$ Gain and NF as function of the signal 1,05µm 1,9µm 0,8µm 0,48µm wavelength. The pump powers are: ■ 400mW, ● 300mW and ▲ 150mW.

•Komukai and co-workers, IEEE J.Quant. Electr. 31, 1880 (1995).

•Aozasa and co-workers, Elect. Lett. 37, 1157 (2001).

II.3 REDG for Fiber Lasers and amplifiers





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800nm+1050nm Pump Scheme for TDFA



FB2 9:15am POST-DEADLINE OFC 2002

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

<u>800nm</u>

GSA – Populates directly the higher amplifying level

<u>1050nm</u>

ESA – Depopulates the lower level + populate the higher level

(12)	Patent Gomes et	Application Publicat	ion	(10) Pub. (43) Pub.	No.: US 2 Date:	2003/02. Dec.	31380 A1 18, 2003		
(50)	9 METHODS AND ARRANGEMENTS IN A			Publication Classification					
PUMPED FIBER AMPLIFIER (76) Investors: Anderson Stevens Localdas Gomes, Recife (IIR); Michael Lee Sundheimer, Resile-FE (IIR), Marias Tarres Carvalito, Canazzafor (IIR);		THER AMPLIFIER Anderson Stevens Lacoldas Gomes, Recife (IIR); Michael Lee Sandheimer, Recife-PE (IIR), Mariana Inrees Carvallos, Caluaragibe (IIR);	(51) Bel, CL ² 10085 M (52) U.S. CL 309(341.3; 59)(341.33; 359(341 (57) ABSTRACT						
	Joagnins Ferreira Martine-Filho, Beciri (SR), Carmolo Jane Albaner Bastos-Filho, Rocle (IR), Watter Marguik, Huddingr (SE) Compositions Address: JEMERS & GILCHRIST, PC 1445 ROSS AVENUE		An i dopt sche inch	An arrangement and method provide an optical thuise doped fiber anophiler utilizing a that wavelength pumping schemes for a smplifying an optical signal. The metho includes the sizes of a find deposition (s) of energy into (s) these methods be assessive wheth realizing of a feet wave					
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(21)	Appl. No.: Filed	18/385,876 Mar. 10, 2003	leng indu	length. The radiation of the first wavelength is arranged to induce, by single photon absorption, a population to the ⁷ H.					
~	Rela	ted U.S. Application Data	wave	elength primar eption of a sin	ily depopulati gle photon, p	es the 3F, b	wel, by success strong encites		
(60)	Provisional 11, 2002. I filed on Ma	application No. 60/363,438, filed on Mar. Provisional application No. 60/365,133, z. 14, 2002.	atala izve pow	state absorption to the ¹ T ₂ level. The steps gives a populatio inversion between the ¹ H ₂ and the ¹ T ₂ levels and facilitate power efficient high gain amplification.					
	2222	120 140		160	150	170			
	100		· · · · ·				-		



Results for the 800+1050nm pumping scheme







Fiber Amplifiers







Fiber Amplifiers





REDG Planar and Channel Waveguides

LINDO.

PLANAR WAVEGUIDE FABRICATION FACILITIES DEPARTAMENTO DE FÍSICA - UFPE

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

Preparation methods employed:

- Proton exchange and Ti-indifusion for LiNbO₃
- Ion Exchange for silicate glasses
- Nonmetalic indifusion 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 Nd³⁺ ion. Switching times around 410 μ s at a repetition rate of 1 kHz was demonstrated. © *1995 American Institute of Physics*.

Appl. Phys. Lett. 66 (4), 23 January 1995



Ion exchange, 8mm long, 2-10 μ m width, $\Delta n = 8.7 \times 10^{-3}$ Silicate glass, 16%Na₂O, 2%Nd₂O₃, K⁺ \leftrightarrow Na⁺





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 $\sim 1m$ 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 (410 μ s). Pump Power dependence: Linear Estimated n₂ ~10⁻¹³ cm²/W

II.4 REDG planar and channel waveguides

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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





C-band waveguide amplifier produced by femtosecond laser writing

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



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

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

- \bullet 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
- > 10⁶ distinguishable possibilities



Rare earth-doped glass microbarcodes

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



Fig. 2. Background fluorescence of RE-doped glasses relative to a bare microscope slide.

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 predetermined 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 μ m thick, 100 μ m wide). The ribbon fiber was scribed every 20 μ m 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.

microbarcodes





Fig. 3. False-color image of two 100 × 20 µm barcodes (Inset) 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) RE images obtained by using a 420-nm long-pass filter.

