Phosphors for solid state lighting (SSL)

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Photo by Tony Mastres

Research on functional inorganic materials

Batteries	Li
Heterogeneous catalysts	Pd
Lone-pair effects and polar materials	Pb
Magnetism and M–I Transitions	Ru
Phosphors for white solid state lighting	Ce
Semiconductors	Zn
Thermoelectric oxides and intermetallics	Mo

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn

Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu

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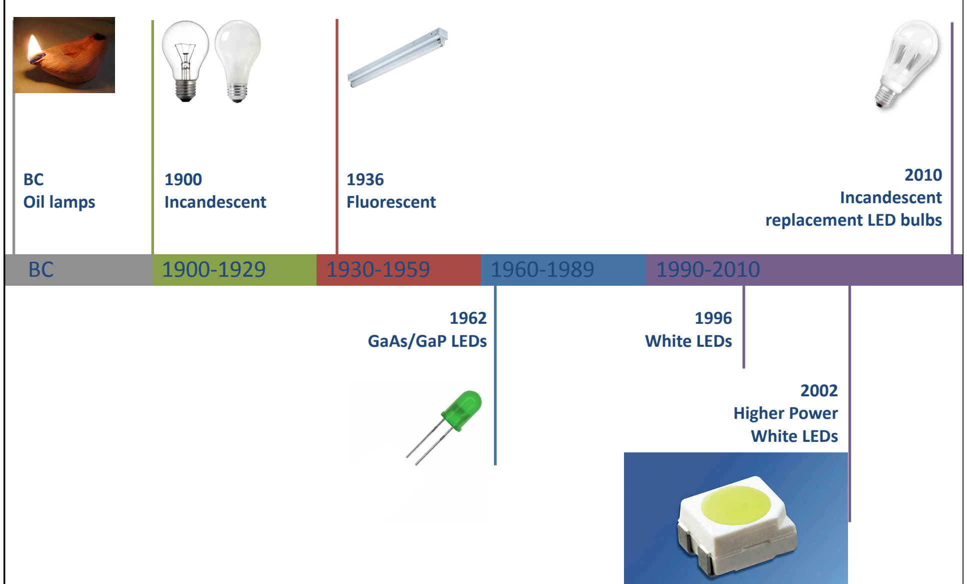
Tutorial on LED Lighting

History of Lighting — Major Innovations



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LED Color Timeline

Conversion Technology

1970
IR to visible up-conversion
red, green, or blue —
for indicator lamps

1996
Partial conversion patents
white and colors —
for, e.g., display backlight
and general lighting

1960 — 1970 — 1980 — 1990 — 2000

Chip Technology

1962
GaAs IR diodes
non-visible
infrared —
for sensors and
remote controls

1965-1975
GaP LEDs
red, amber, or green —
for indicator lamps

1993
Bright GaN
blue LED

Comparison to Other Types of Lighting

Incandescent Light Bulb

Only 15 lm/W

LED

> 70 lm/W

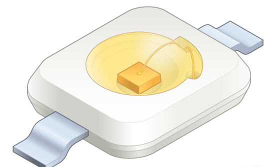
Lumens per Watt (lm/W) measures efficiency

Comparison to Other Types of Lighting

Incandescent Light Bulb

Change light bulb every 850 hours

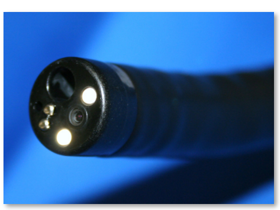
LED



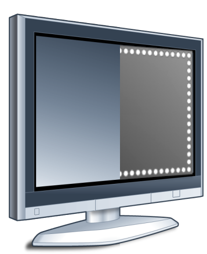
Only change LED every 10,000-50,000 hours

Comparison to Other Types of Lighting

Small Size/Miniature Lamps



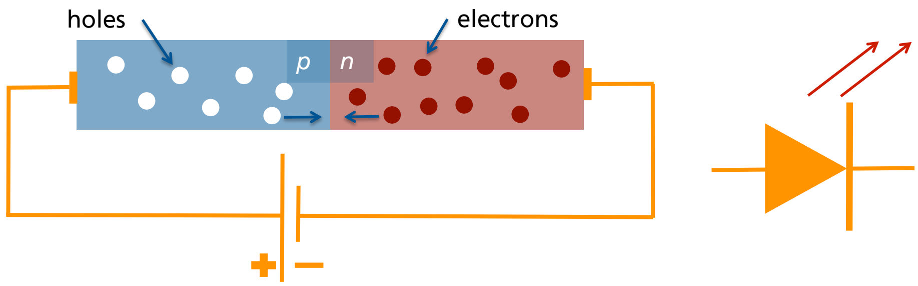
Medical Applications



LCD TVs

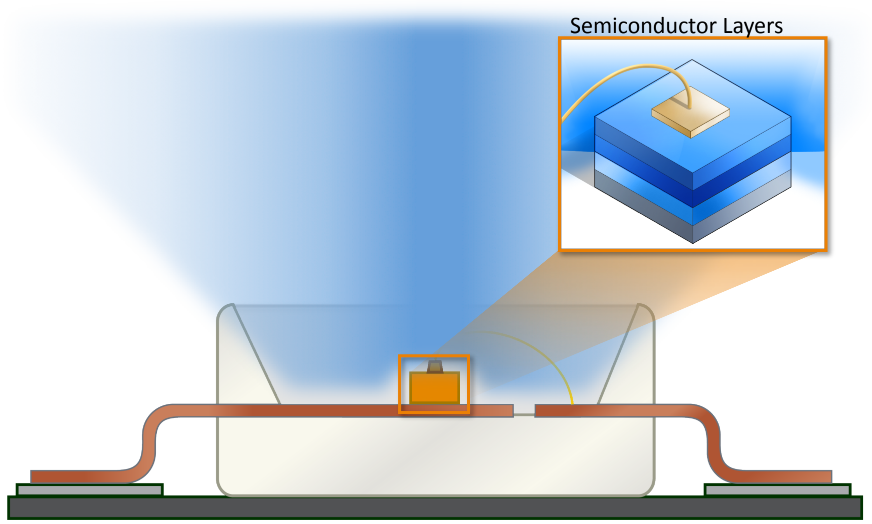
Decorative Lighting

How do LEDs work?



- Semiconductor *p-n* junction
- Subject to forward bias, which creates pairs of electrons and holes (excitons)
- Delocalized excitons recombine to emit light

How do LEDs work?



White light

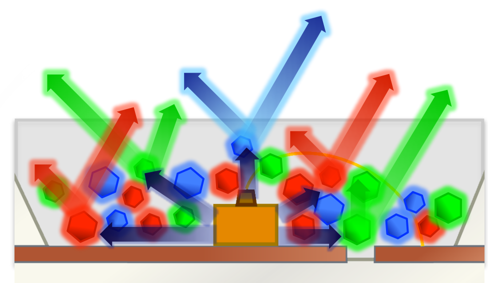
- White light does not exist as a spectral color
- Polychromatic light, *e.g.*, white light, can be created by mixing colors
- The mixed color produced depends on the ratio of the source color

3-LED solution

What is holding this back?

- Needs 3 power supplies
- Expensive
- LED intensities need to be readjusted for temperature changes and burning time

Full conversion



LED with red, green, and blue phosphors

Fluorescent lamp

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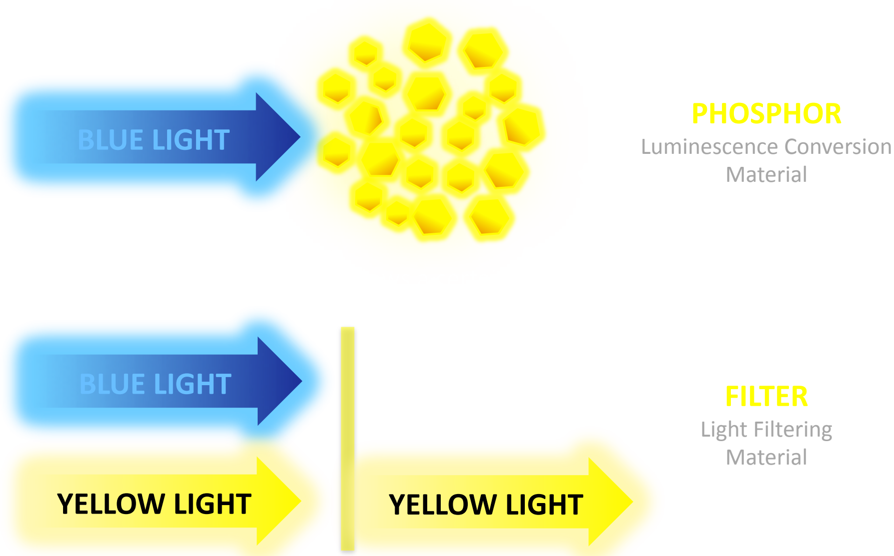
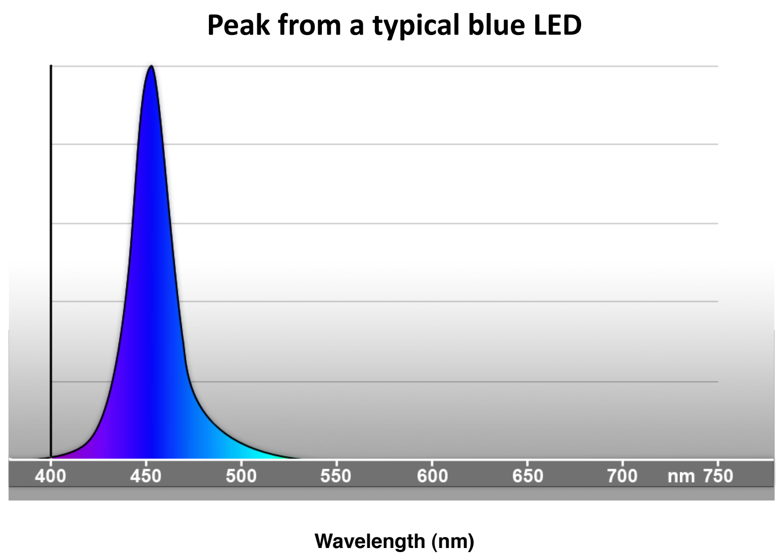
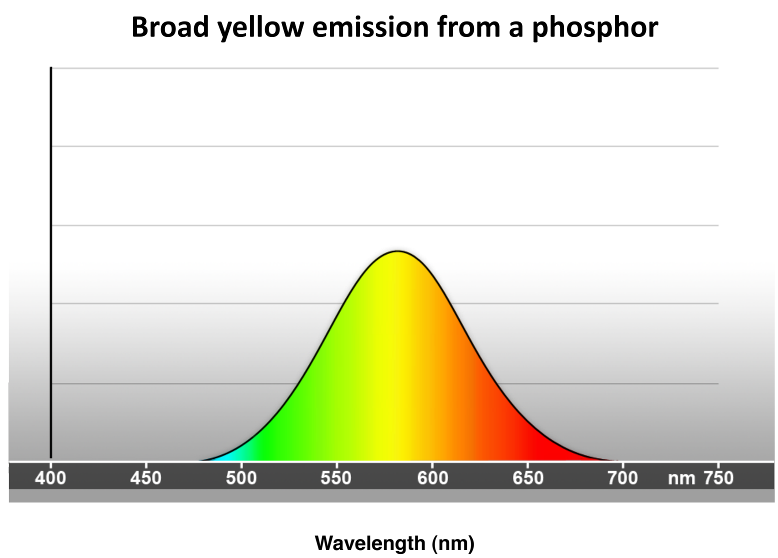
Partial conversion: The solution of choice



Partial conversion: The solution of choice

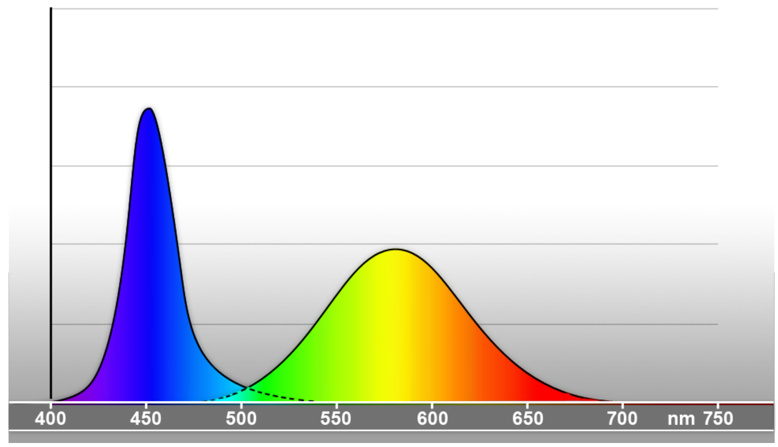


Partial conversion: The solution of choice



Partial conversion: The solution of choice

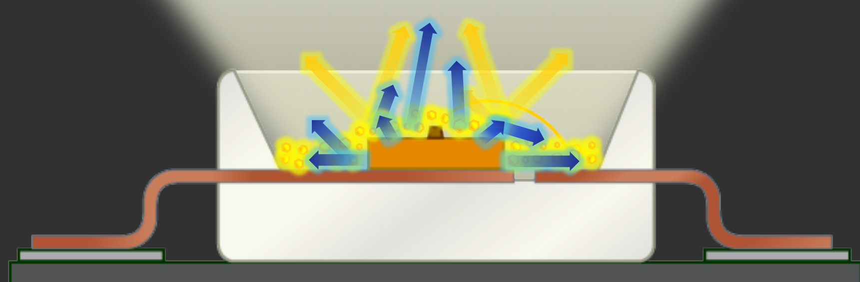
White light created by partial conversion



Wavelength (nm)

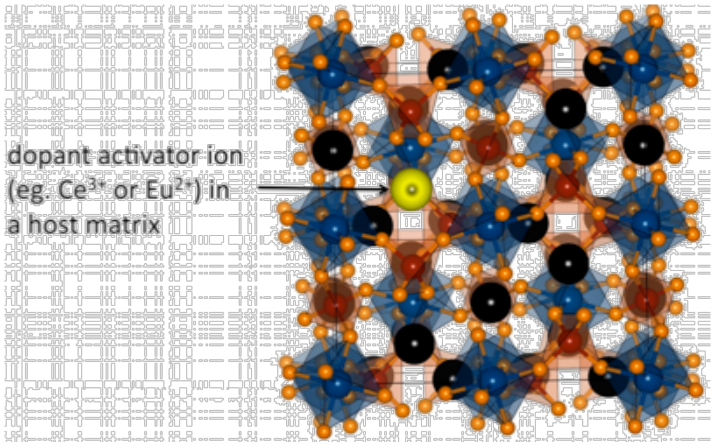
Partial conversion: The solution of choice

- Some of the blue light from the LED is converted
- Some of the blue light is scattered
- The two combine to create a third color (e.g., white light)

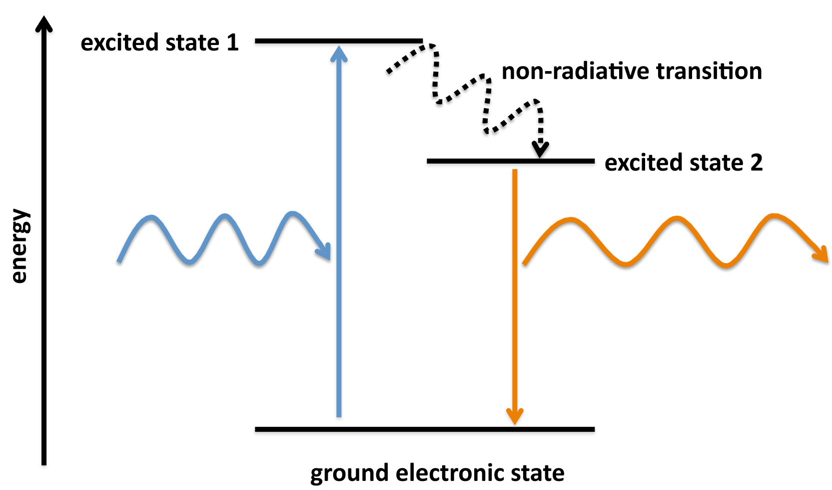


How is partial conversion achieved: Phosphors

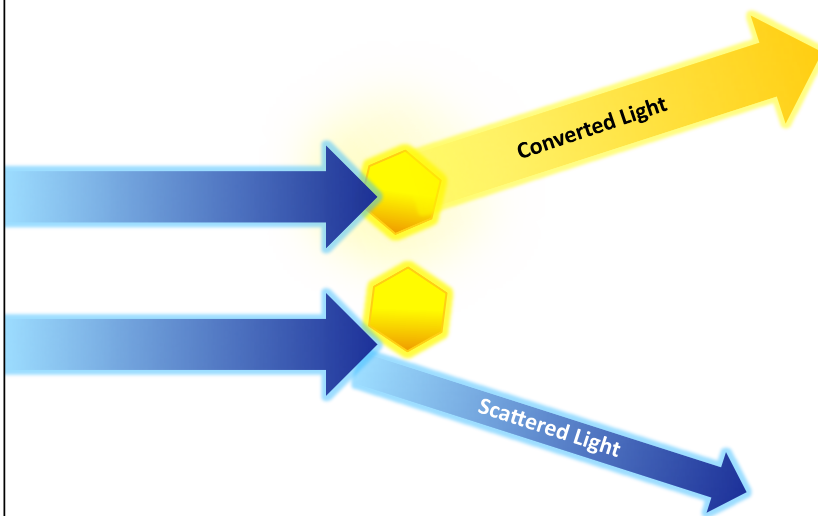
- Electrons in the activator ion absorb incident light and re-emit at longer wavelengths



The workings of a phosphor

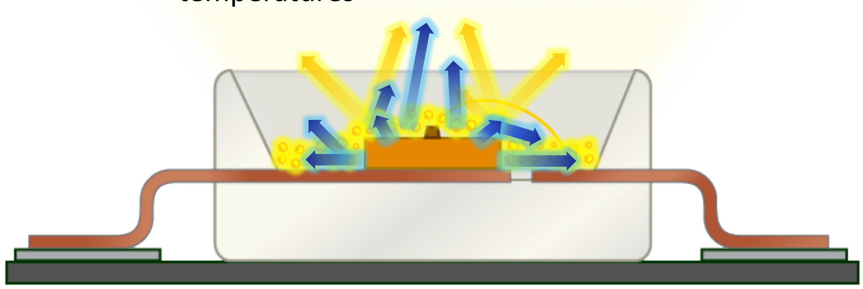


The workings of a phosphor (roles as scatterer)



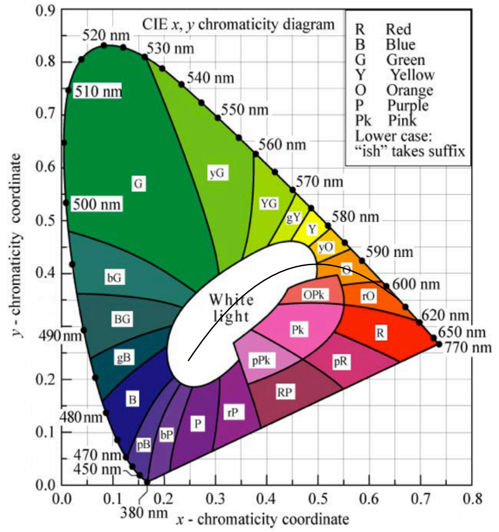
Inorganic phosphors

Advantages:
- Does not degrade
- Light scattering by inorganic powders greatly reduces light trapping
- Much better performance at elevated temperatures



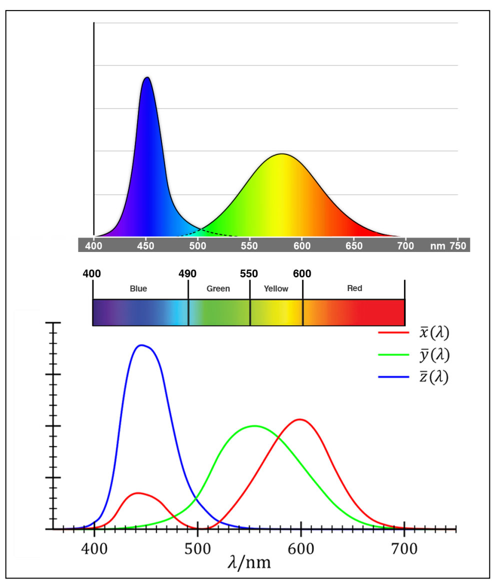
The CIE Diagram

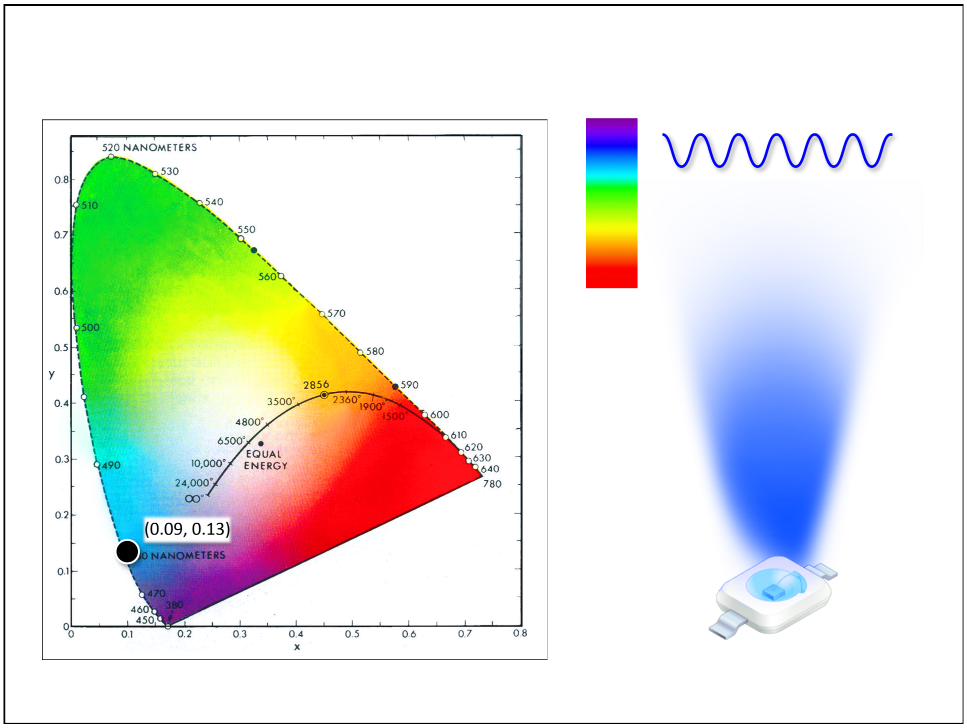
- Shows all light colors in an x, y coordinate system
- Pure spectral colors lie along the border
- Polychromatic light lies within the interior
  - a mixture of spectral colors



Obtaining the CIE coordinates

- Experimental spectrum is fit to tristimulus function displayed below on the right
- Three values obtained, of *x*, *y*, and *z*, that are constrained according to $x + y + z = 1$

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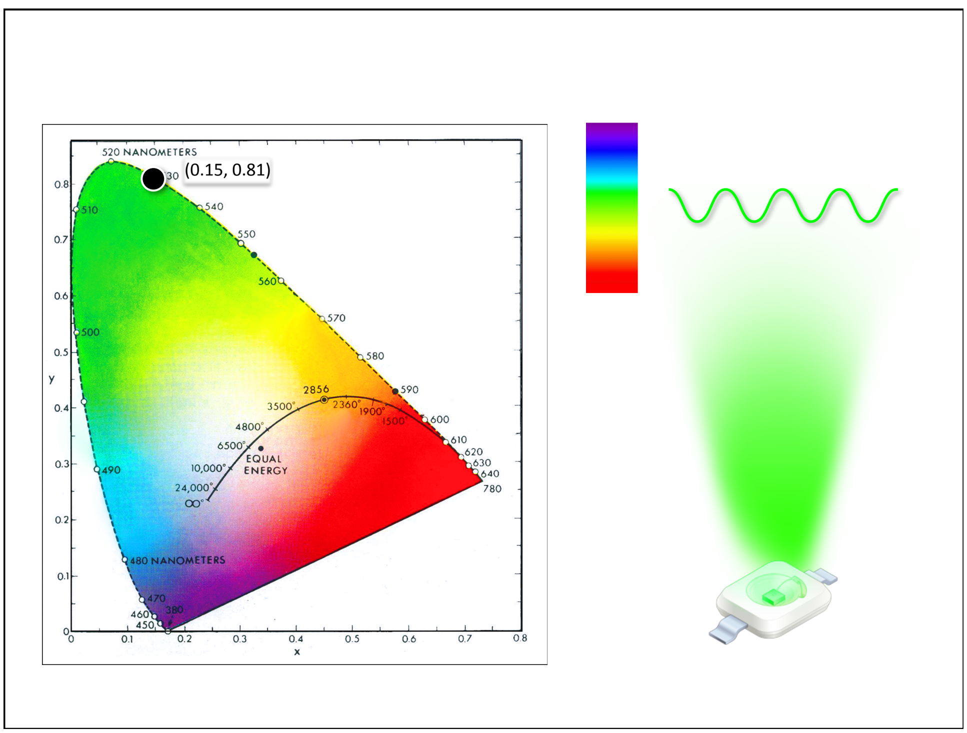
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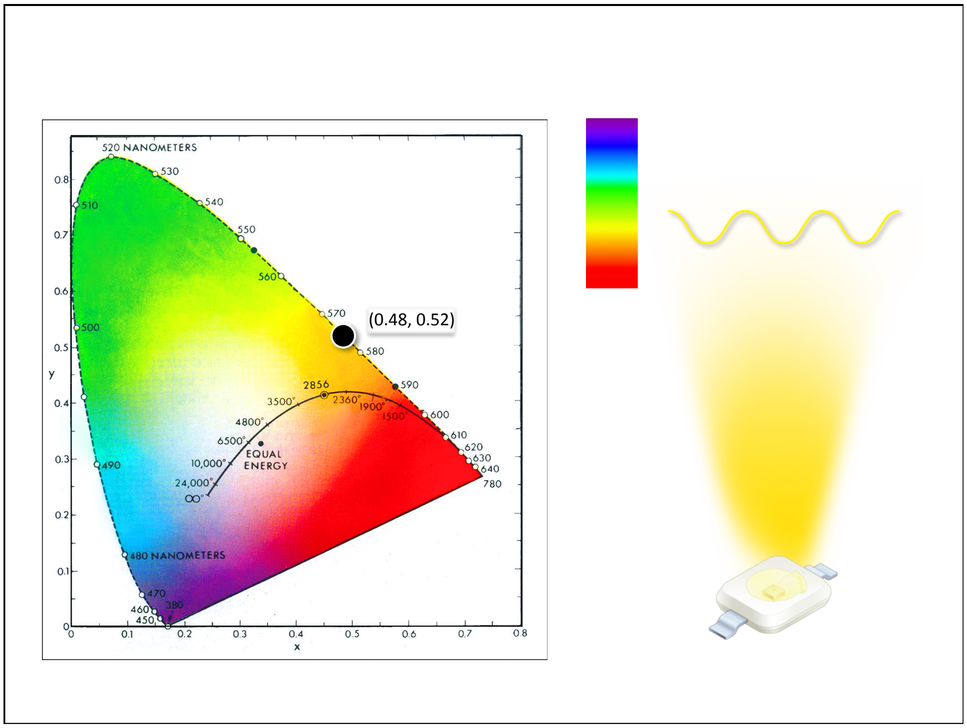
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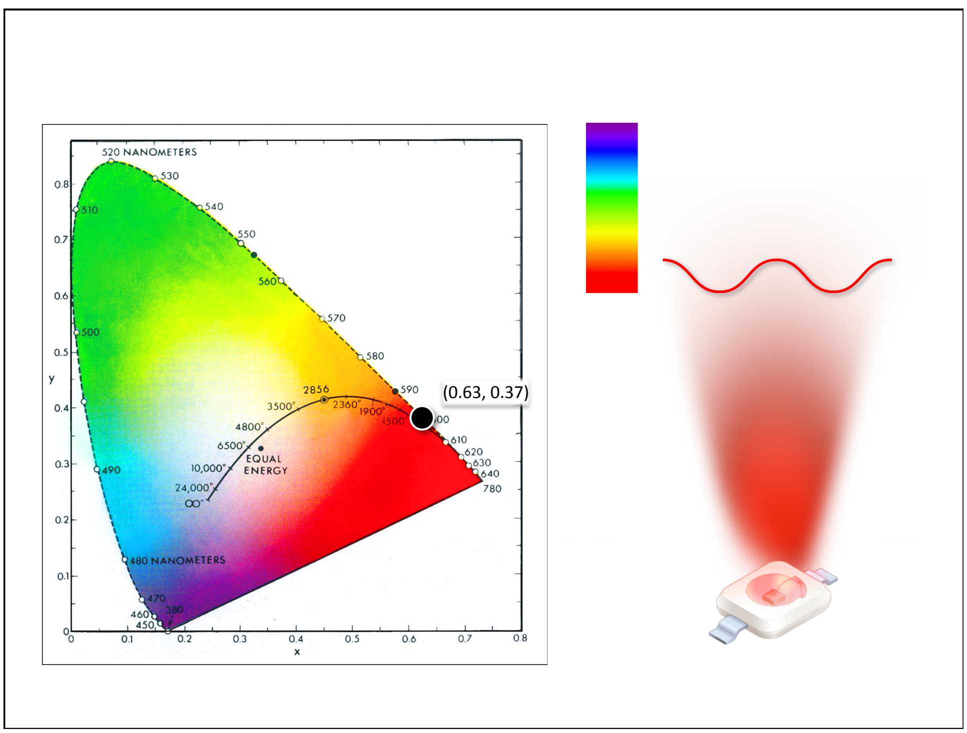
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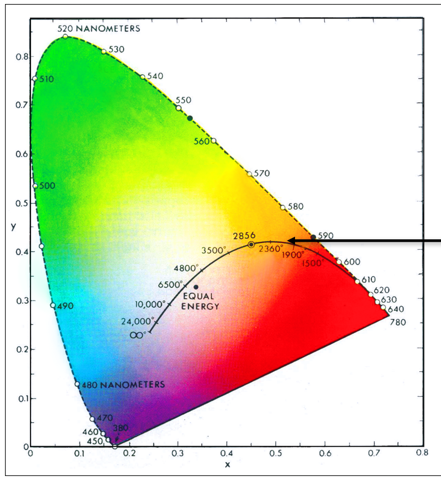
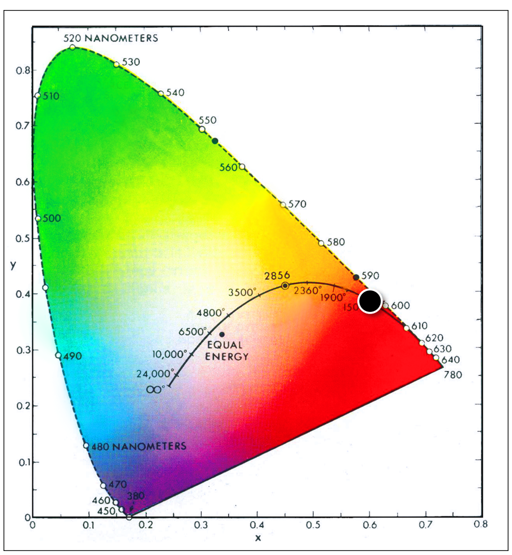
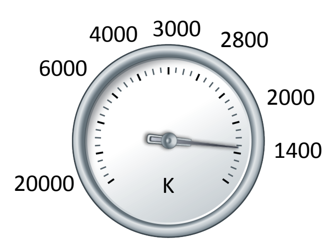
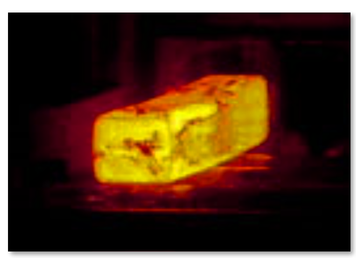
- Planckian radiators define polychromatic light in certain areas of the CIE diagram

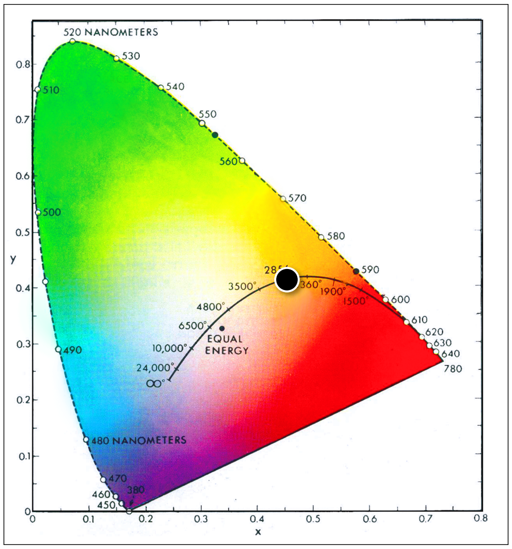


Locus of Planckian radiators





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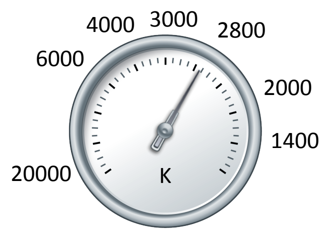
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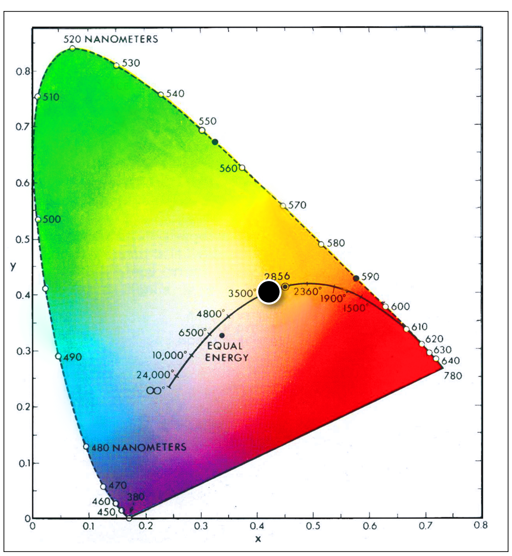
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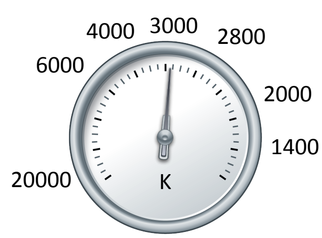
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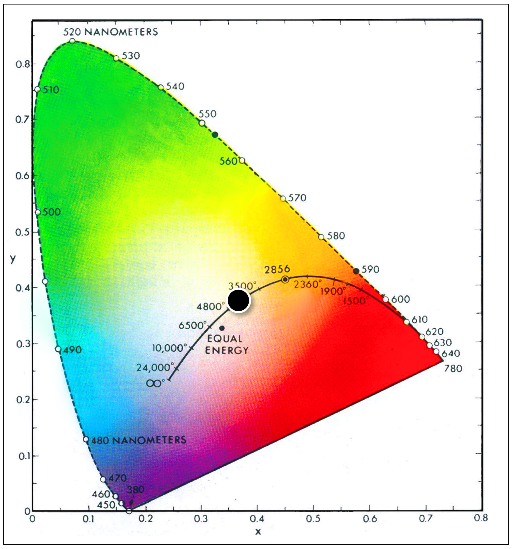
Warm White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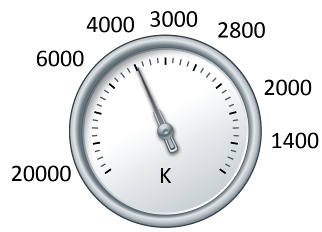
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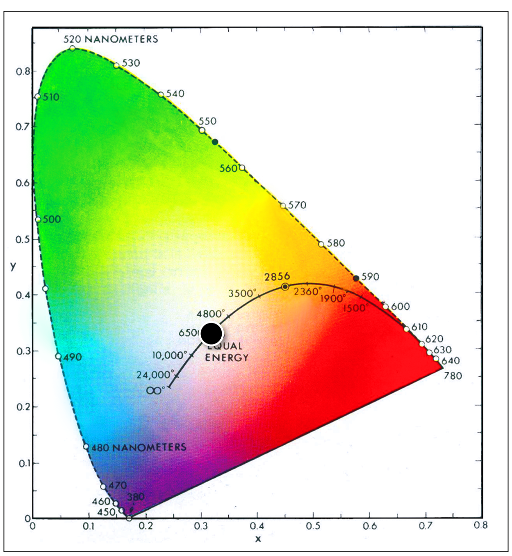
Cool White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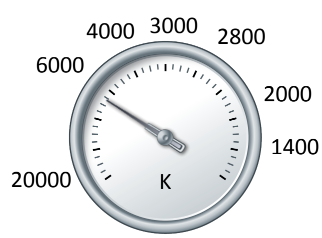
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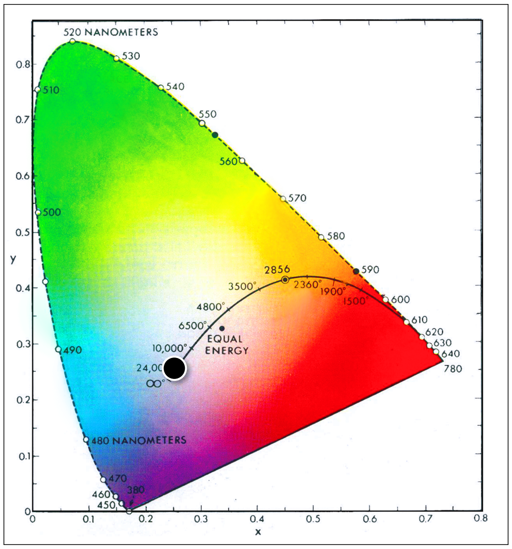
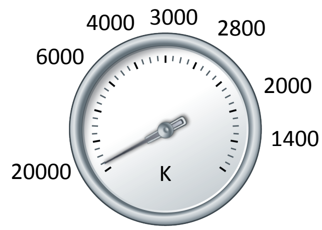
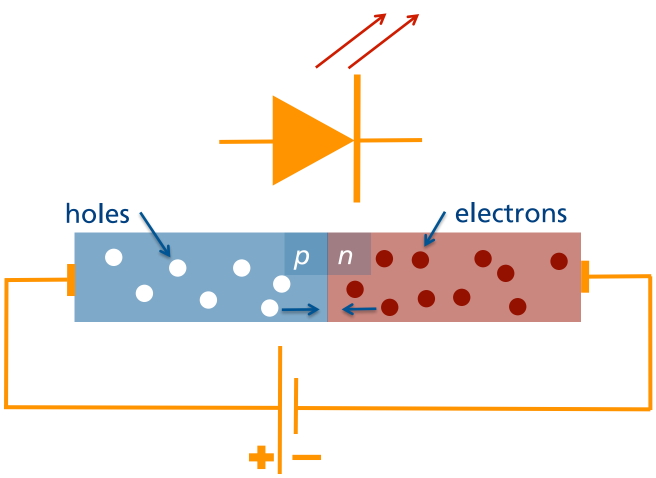
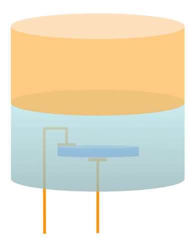




LEDs: Converting electricity to light (not heat !)





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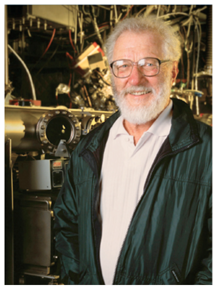
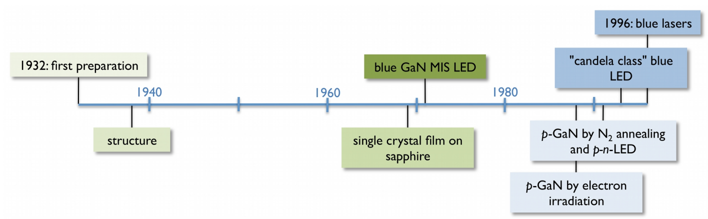
LEDs: Converting electricity to (white) light

A history closely allied with two UCSB researchers:



Herb Kroemer (2000 Nobel) Heterostructures, laser diodes …

Shuji Nakamura (2011 Emmy) GaN lighting (blue LEDs), blue laser diodes … materials *chemistry of GaN*



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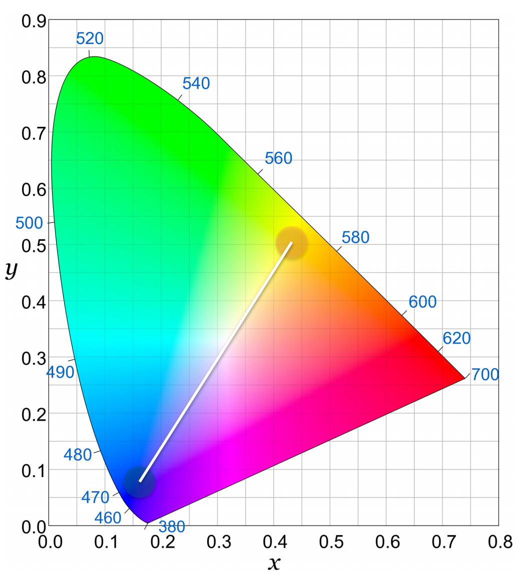
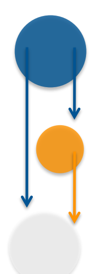
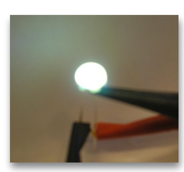
Solid state lighting strategies



1. Tricolor LEDs with no need for phosphors
2. UV LED + RGB phosphors
3. Blue LED + yellow/orange phosphors



Blue LED (450 nm)



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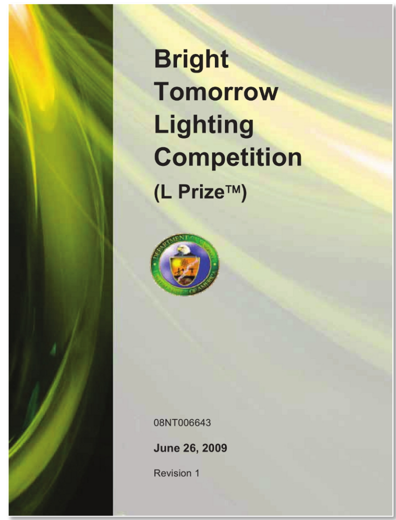
The L Prize

60 W Incandescent Replacement Lamp
- More than 90 lm/W
- Less than 10 watts
- More than 900 lumens
- More than 25,000 hour life
- More than 90 CRI
- Between 2700 K – 3000 K CCT



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Some applications of solid state lighting

Audi A4

The LS 600h L is the world's first vehicle to be equipped with Light-Emitting Diode (LED) headlamps for low-beam use…a subtle blue tint, giving this LS a more modern appearance.

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Some applications of solid state lighting

The Chanel Building in Osaka (Peter Marino Architect).

LED lamps for indoor cultivation

90% Less Heat
50,000 Hour Rated
LEDs Made in USA
No Heat Signature
Cut Your Electricity Bill

High Times Magazine

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Ce^{3+} phosphors for solid state white lighting

The need: A means of efficiently converting blue light from InGaN LEDs to white light with high color rendition.

The task: Understand how known Ce^{3+} phosphors work, and develop new hosts that outperform current ones. Explore known structure types *via* compositional tuning.

Requires tools to understand local structure around Ce^{3+} of phosphors.

People involved:

Professor Steve DenBaars, Solid State Lighting and Energy Center
Dr. Won Bin Im (Chonnam National University, Korea)
Nathan George, Kristin Denault, Dr. Alex Birkel, Dr. Jakoah Brgoch, Michael Cantore, Leah Kuritzky, Stuart Brinkley
Dr. Alexander Mikhailovsky

Dr. Kate Page, Anna Llobet (Los Alamos, LANSCE)
Dr. Mahal Balasubramanian (APS, Argonne)

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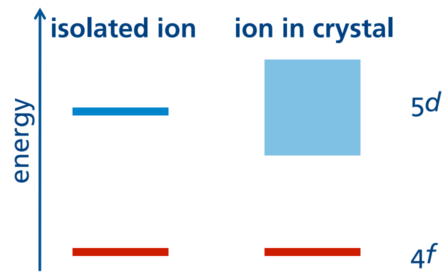
Understanding Ce^{3+} phosphors (similar in Eu^{2+})

Ce is $[Xe]4f^15d^16s^2$ Ce^{3+} is $[Xe]4f^15d^06s^0$ Ce^{4+} is $[Xe]4f^05d^06s^0$

The transition from 4*f* to 5*d* in Ce^{3+} is spin allowed – it is narrow and takes place in the UV in isolated Ce^{3+} atoms.

In solids, the crystal field broadening of the 5*d* states allows absorption in the near-UV/blue, and emission in the visible.



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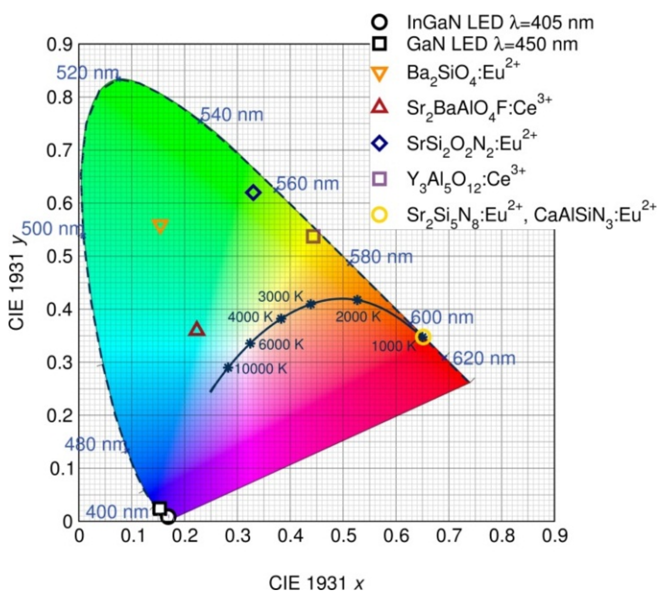
Solid state lighting: Near-UV or blue LED + yellow phosphor



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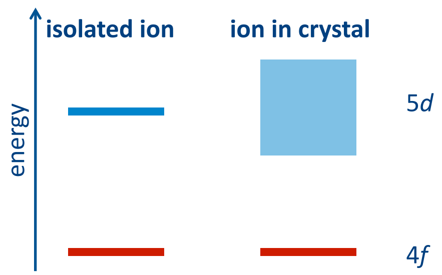
The transition from 4*f* to 5*d* in Ce^{3+} is spin allowed – it is narrow and takes place in the UV in isolated Ce^{3+} atoms.

In solids, the crystal field broadening of the 5*d* states allows absorption in the near-UV/blue, and emission in the visible.



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1. New oxyfluoride phosphors

2. Are there better, quicker ways of making known phosphors?

3. What makes some phosphors efficient and others not: Hints from thermal quenching behavior, and displacement ellipsoids.

4. Probing the local structure around the activator.

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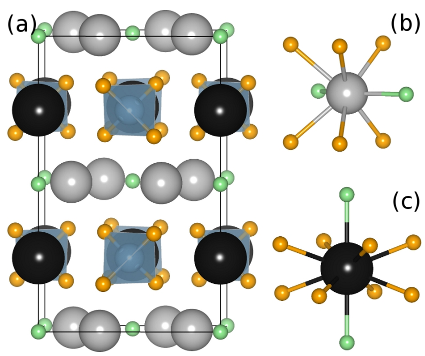
Other research: New oxide and oxyfluoride phosphors

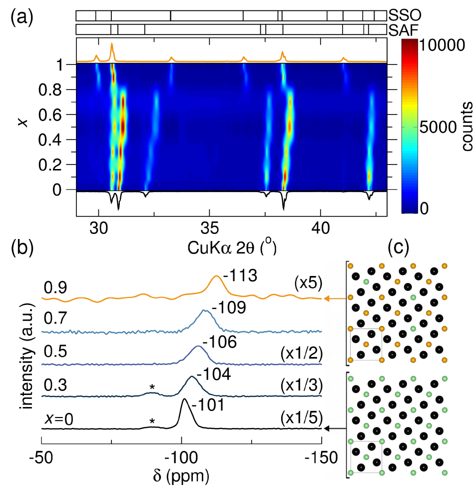
I4/mcm structure of $LaSr_2AlO_5$ and Sr_3AlO_4F



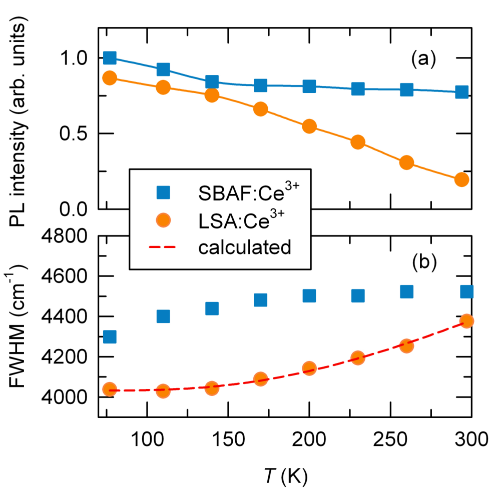
$LaSr_2AlO_5$ ⟷ Sr_3AlO_4F

Im, Brinkley, Mikhailovsky, Hu, DenBaars, Seshadri, *Chem. Mater*. **22** (2010) 2842–2849; Im, George, Kurzman, Brinkley, Mikhailovsky, Hu, Chmelka, DenBaars, Seshadri, *Adv. Mater.* **23** (2011) 2300–2305.

$(SAF)_{1-x}(SSO)_x:Ce^{3+}$ solid solutions.



New oxyfluoride phosphors

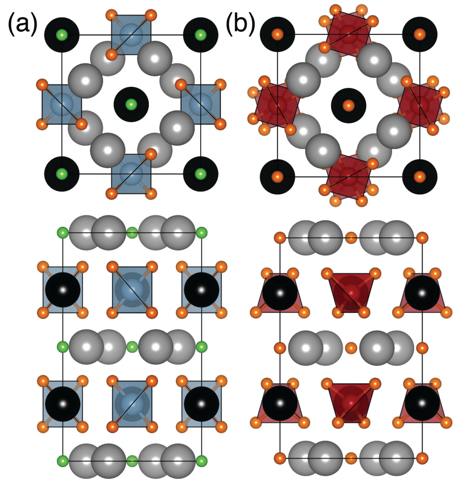


Thermal quenching properties are greatly improved over our old phosphor, $LaSr_2AlO_5$: comparable with YAG:Ce^{3+}

So what is the problem?

Stability to water is poor!

New oxyfluoride phosphors



Solid solution between Sr_3AlO_4F and Sr_3SiO_5: $(SAF)_{1-x}(SSO)_x$:Ce^{3+}

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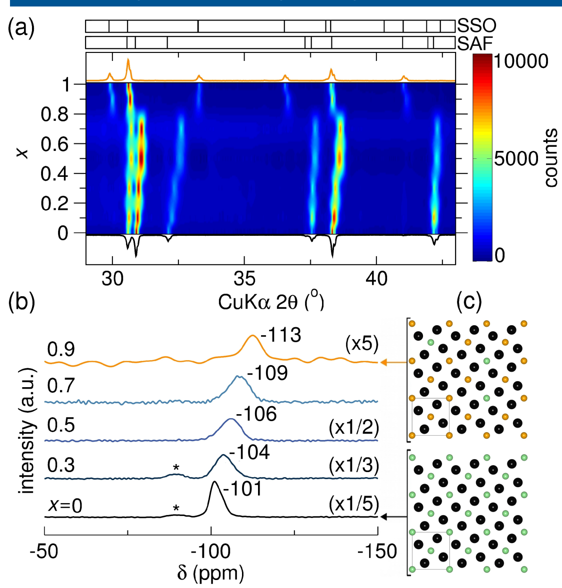
New oxyfluoride phosphors



$(SAF)_{1-x}(SSO)_x$:Ce^{3+}

XRD and ^{19}F NMR confirm the solid solution.

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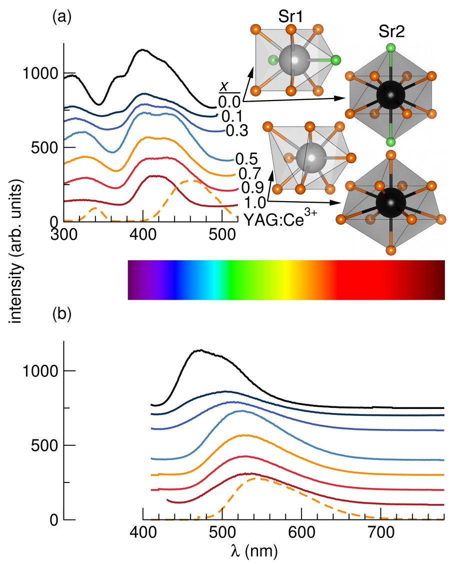
New oxyfluoride phosphors

$(SAF)_{1-x}(SSO)_x:Ce^{3+}$



The solid solution ensures more stable compounds as well as more red-shifted absorption and emission.

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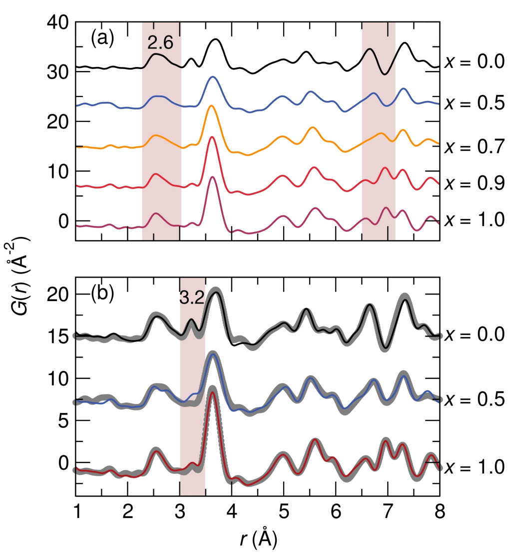
New oxyfluoride phosphors

$(SAF)_{1-x}(SSO)_x:Ce^{3+}$



X-ray pair distribution functions (APS 11-ID-B) allow some understanding of local structural changes.

The highest quantum yields are near $x = 0.5$

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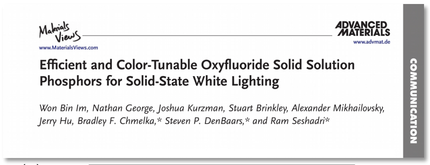
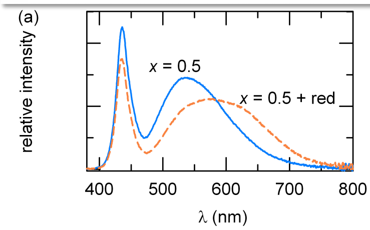
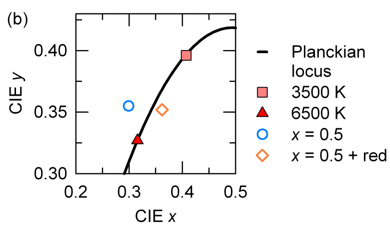
New oxyfluoride phosphors


Efficient and Color-Tunable Oxyfluoride Solid Solution Phosphors for Solid-State White Lighting

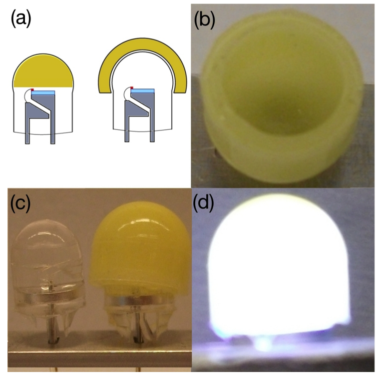
Won Bin Im, Nathan George, Joshua Kurzman, Stuart Brinkley, Alexander Mikhailovsky, Jerry Hu, Bradley F. Chmelka,* Steven P. DenBaars,* and Ram Seshadri*





$(SAF)_{1-x}(SSO)_x:Ce^{3+}$

Color and efficiency are good. "Phosphor cap" strategy helps increase efficacy greatly.



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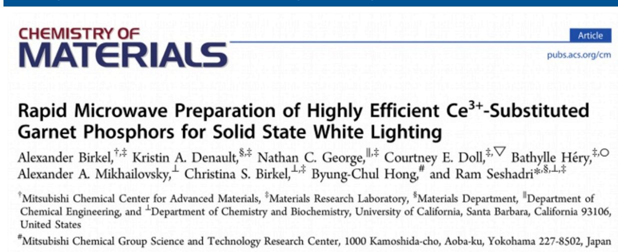
A quicker route to phosphors: The use of microwaves


CHEMISTRY OF MATERIALS

Article

pubs.acs.org/cm

Rapid Microwave Preparation of Highly Efficient Ce^{3+}-Substituted Garnet Phosphors for Solid State White Lighting

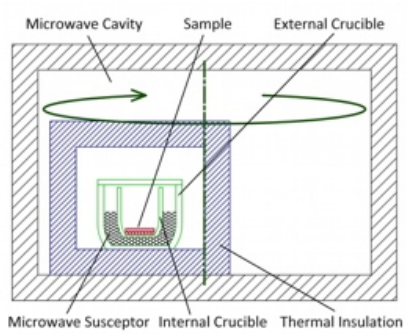
Alexander Birkel,[†,‡] Kristin A. Denault,[§,‡] Nathan C. George,[∥,‡] Courtney E. Doll,[‡,▽] Bathylle Héry,[‡,○] Alexander A. Mikhailovsky,[⊥] Christina S. Birkel,[⊥,‡] Byung-Chul Hong,[#] and Ram Seshadri[*,§,⊥,‡]

[†]Mitsubishi Chemical Center for Advanced Materials, [‡]Materials Research Laboratory, [§]Materials Department, [∥]Department of Chemical Engineering, and [⊥]Department of Chemistry and Biochemistry, University of California, Santa Barbara, California 93106, United States

[#]Mitsubishi Chemical Group Science and Technology Research Center, 1000 Kamoshida-cho, Aoba-ku, Yokohama 227-8502, Japan

With Byung-Chul Hong, Mitsubishi Chemicals.

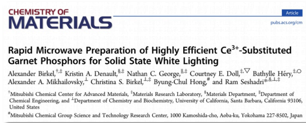
No coupling when cold. Carbon susceptor ensures initial coupling, and reducing conditions.



Birkel *et al.* Chem. Mater. **24** (2012) 1198–1204.

UC SANTA BARBARA science & engineering

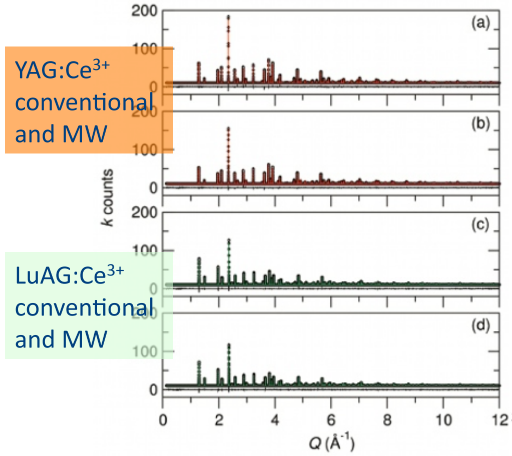
A quicker route to phosphors: The use of microwaves

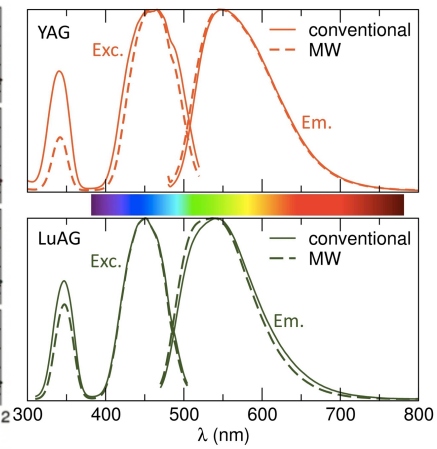

CHEMISTRY OF MATERIALS

Rapid Microwave Preparation of Highly Efficient Ce^{3+}-Substituted Garnet Phosphors for Solid State White Lighting

Alexander Birkel, Kristin A. Denault, Nathan C. George, Courtney E. Doll, Bathylle Héry, Alexander A. Mikhailovsky, Christina S. Birkel, Byung-Chul Hong, and Ram Seshadri

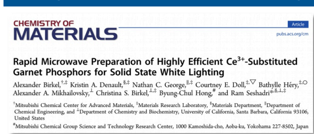
Synchrotron x-ray studies establish completely clean phases. Optical properties similar to furnace prepared samples (using 95% less time).





Birkel *et al.* Chem. Mater. **24** (2012) 1198–1204.

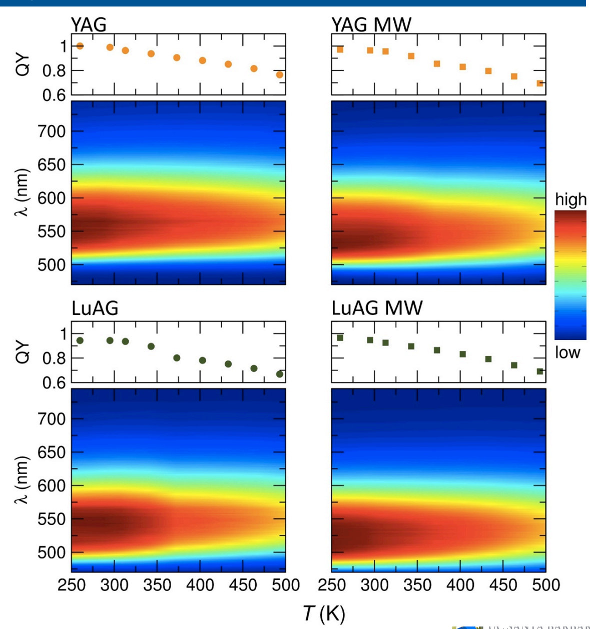
A quicker route to phosphors: The use of microwaves


CHEMISTRY OF MATERIALS

Rapid Microwave Preparation of Highly Efficient Ce^{3+}-Substituted Garnet Phosphors for Solid State White Lighting

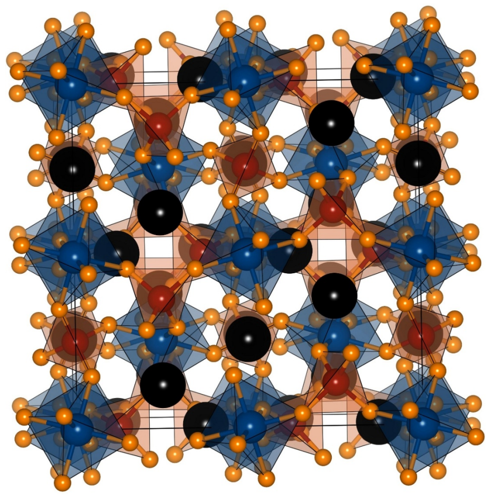
Alexander Birkel, Kristin A. Denault, Nathan C. George, Courtney E. Doll, Bathylle Héry, Alexander A. Mikhailovsky, Christina S. Birkel, Byung-Chul Hong, and Ram Seshadri

T-dependence of emission show similar stability for conventionally-prepared and MW samples.



Birkel *et al.* Chem. Mater. **24** (2012) 1198–1204.

The canonical material: $Y_3Al_5O_{12}:Ce^{3+}$ (Blasse)



Garnet crystal structure: AlO_4 tetrahedra and AlO_6 octahedra, all completely corner-connected.

Y occupies 8-coordinate voids formed by AlO_n polyhedra.

Pyrope	$Mg_3Al_2Si_3O_{12}$
Almandine	$Fe_3Al_2Si_3O_{12}$
Spessartine	$Mn_3Al_2Si_3O_{12}$
Andradite	$Ca_3Fe_2Si_3O_{12}$
Grossular	$Ca_3Al_2Si_3O_{12}$
Uvarovite	$Ca_3Cr_2Si_3O_{12}$

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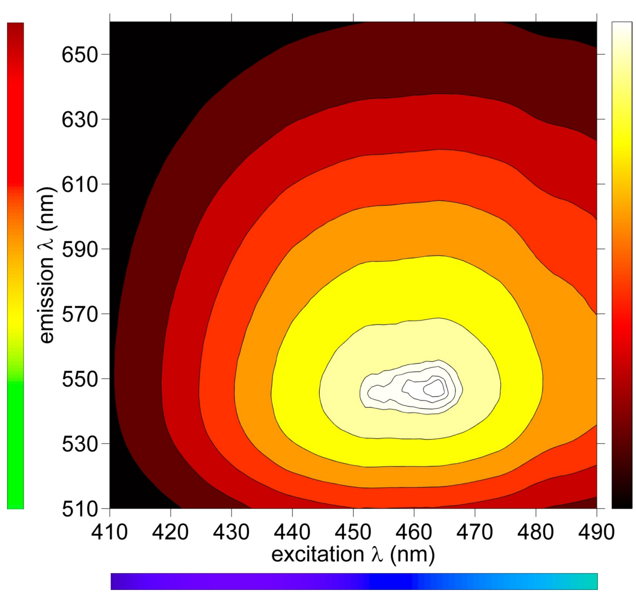
The canonical material: $Y_3Al_5O_{12}:Ce^{3+}$



Excitation and emission of YAG:Ce^{3+} are well-suited to creation of white.

The high refractive index helps with scattering.

The large Stokes shift prevents re-absorption.

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Understanding the canonical phosphor: YAG:Ce^{3+}

All the action is around the very small amount of Ce^{3+} in the lattice: 2% substitution for Y^{3+}. How do we probe this?

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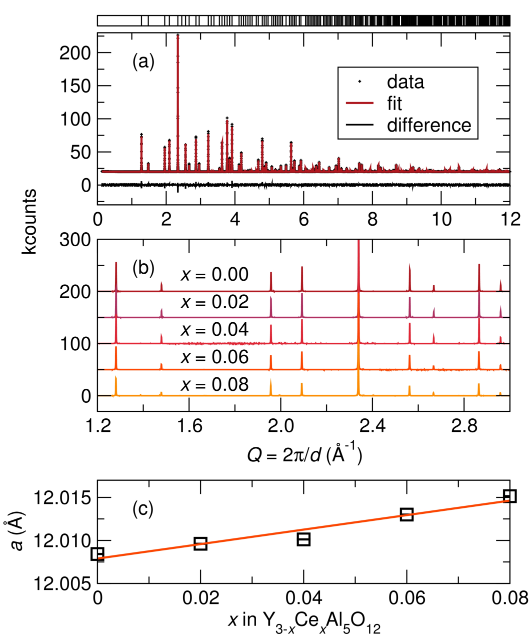
Understanding Ce^{3+} in YAG:Ce^{3+}



Synchrotron X-ray Rietveld refinement (11-BM, Argonne) reveals lattice expansion upon substitution of Y^{3+} by the slightly larger Ce^{3+}.

In $Y_{3-x}Ce_xAl_5O_{12}$, for $x = 0.06$ (optimal), the cell expands by ≈ 300 ppm compared to $x = 0$.

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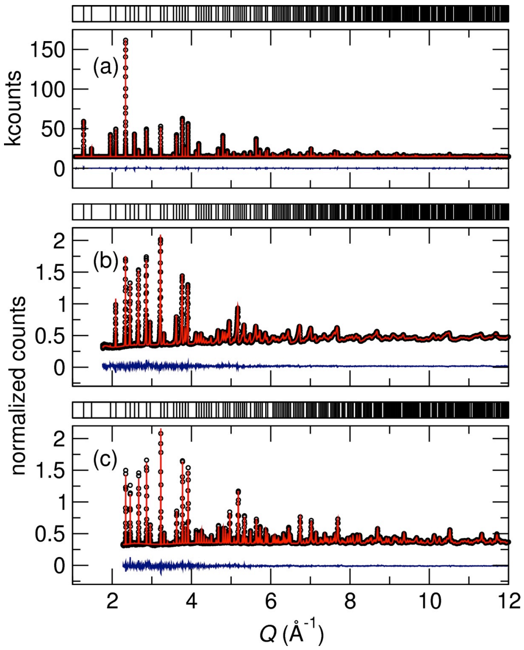
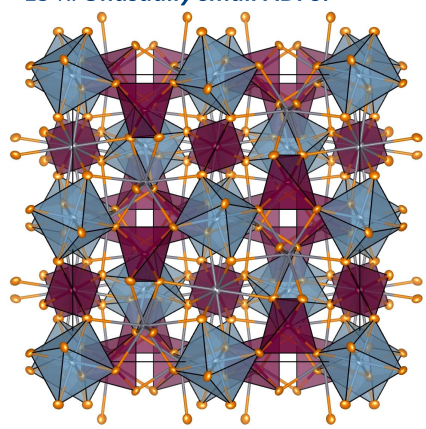
Understanding Ce^{3+} in YAG:Ce^{3+}: Small ADPs stiff lattice



Rietveld refinement of synchrotron X-ray and neutron (HIPD, Los Alamos) data at room temperature and neutron (NPDF, Los Alamos) at 15 K. ***Unusually small ADPs!***



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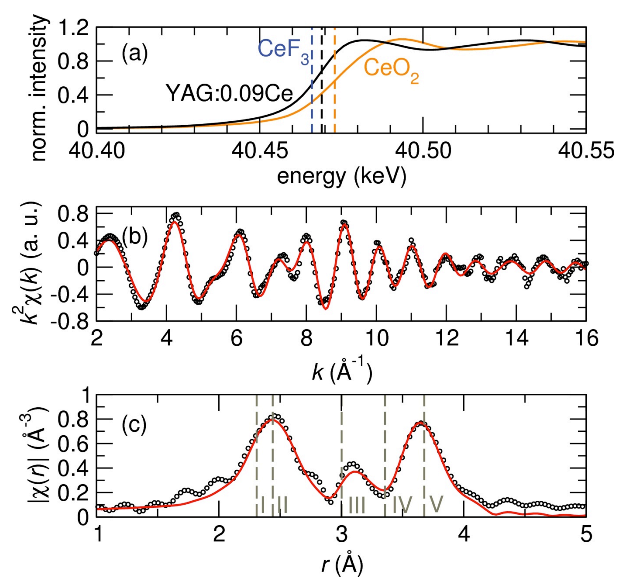
Understanding Ce^{3+} in YAG:Ce^{3+}: Small ADPs stiff lattice



Small Debye-Waller factors confirmed by Ce-K-edge EXAFS.

Also a suggestion of 3% expansion around Ce^{3+} [Cf. Ghigna, Pin, Ronda, Speghini, Piccinelli, Bettinelli, *Opt. Mater.* **34** (2011) 19–22, Ce-L-edge.]

Ce^{3+} coordination relaxes to average by the fifth coordination shell.

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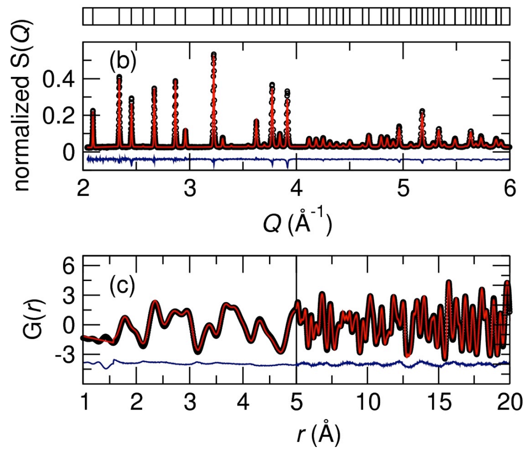
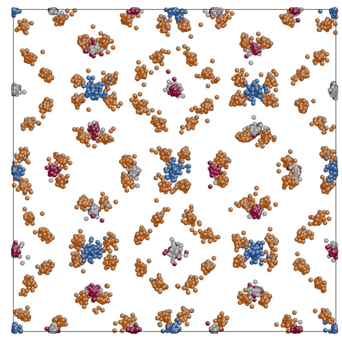
Understanding Ce^{3+} in YAG:Ce^{3+}: Total scattering analysis





RMC analysis of the total neutron scattering (NPDF, Los Alamos) allows the local structure around Ce^{3+} to be *independently* probed of Y^{3+}. Neutrons are more sensitive to oxygen, and in principle, provide better metal-oxygen distances.

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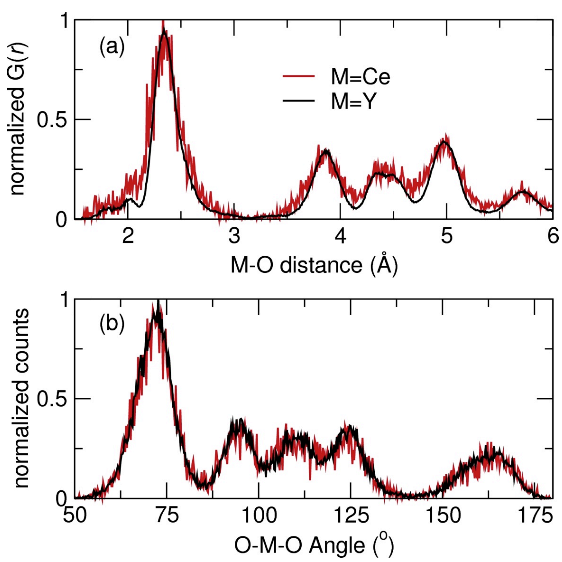
Understanding Ce^{3+} in YAG:Ce^{3+}: Total scattering analysis



Within the resolution of total scattering and RMC, the Ce—O distances are indistinguishable from Y—O distances. Suggests a highly rigid lattice (as do the small ADPs) and Ce^{3+} under high compression.

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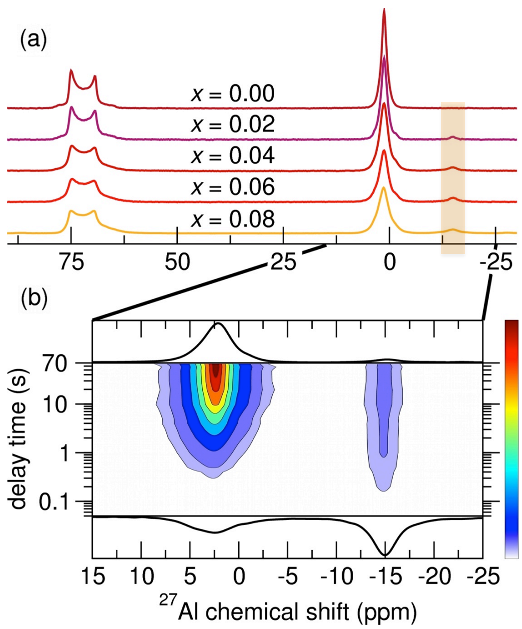
Understanding Ce^{3+} in YAG:Ce^{3+}: ^{27}Al MAS NMR

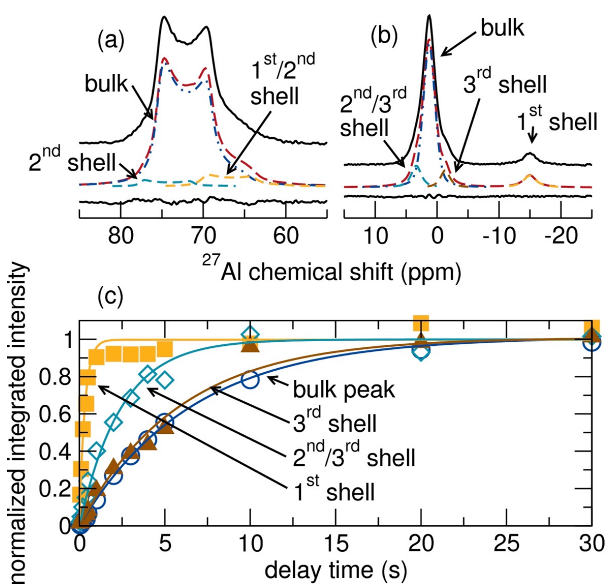


800 MHz (19.6 T) NMR spectrometer at UCSB.

New features in the ^{27}Al NMR upon Ce^{3+} substitution: Ce^{3+} as an internal contrast reagent.

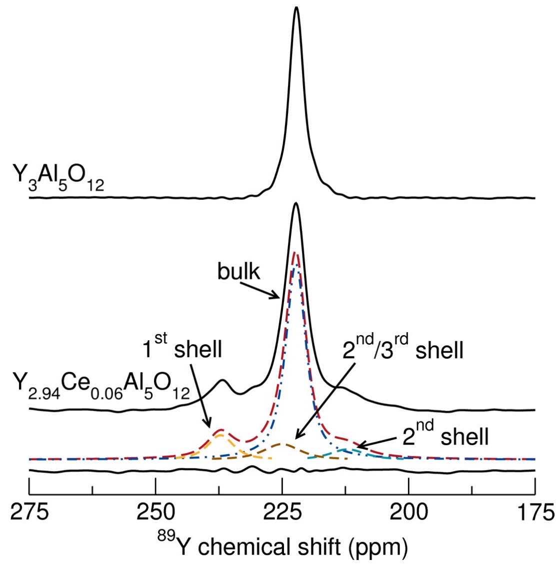
Saturation recovery experiments confirm that the new features correspond to ^{27}Al proximal to paramagnetic Ce^{3+}.

Understanding Ce^{3+} in YAG:Ce^{3+}: ^{27}Al MAS NMR



The new features can be quantitatively fit to different shells around the small amounts of Ce^{3+} substituents.

Understanding Ce^{3+} in YAG:Ce^{3+}: ^{27}Al MAS NMR



^{89}Y NMR similarly displays new features upon Ce^{3+} substitution, that can also be accounted for by the paramagnetism of Ce^{3+}

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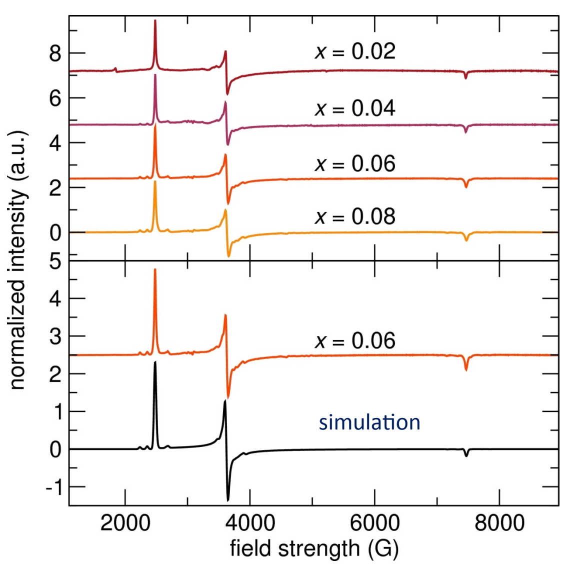
Understanding Ce^{3+} in YAG:Ce^{3+}: ^{27}Al MAS NMR



ESR at 4.2 K directly probes the Ce^{3+}. The ESR spectra can be modeled with some antisite disorder, (Y^{3+} in octahedral Al^{3+} site) of the order of 2%, that is confirmed by the neutron refinement.

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Understanding Ce^{3+} in YAG:Ce^{3+}: Findings

YAG unit cell expands slightly with increasing amounts of Ce (about a 300 ppm increase from $x = 0$ to $x = 0.06$).

Ce K-edge XANES and ^{27}Al NMR results show that all the Ce is reduced to Ce^{3+}.
Ce K-edge EXAFS reveals a 3% expansion in Ce-O bond distance compared to the average Y-O distance, that relaxes by the 5th coordination shell (3.7 Å). RMC/total neutron scattering simulations in agreement.

Analysis of ADPs and calculation of Θ_D confirms that the YAG lattice is very rigid, with few accessible phonons available at LED operating temperatures.

^{27}Al and ^{89}Y NMR experiments also show that the unpaired 4*f* electron in Ce^{3+} causes a displacement in the NMR signal of nearby nuclei, as well as a greatly shortened T_1 relaxation time of nearby nuclei.

EPR spectra of the YAG:Ce phosphor show small satellite signals around the main absorption signal, corresponding to Y-Al(oct.) antisite defects in YAG:Ce of around 2 mol % of octahedral Al sites.

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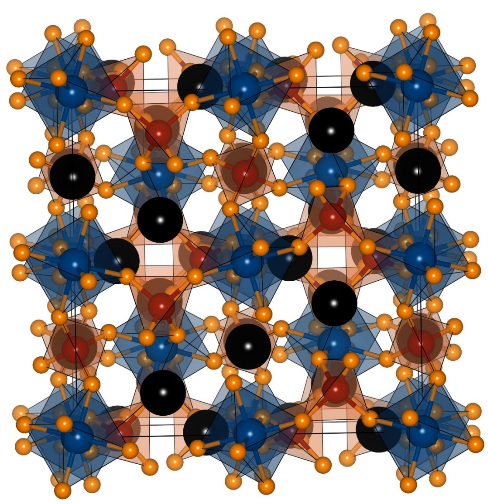
Understanding Ce^{3+} in YAG:Ce^{3+}: Why is it canonical?



Rigid 3D connectivity – a consequence of low charge on Al^{3+} [Pauling's rules of crystal chemistry].

Large band gap because of Al, and connectivity.

Stiff lattice because of connectivity and light elements: Also low quenching (incl. thermal).

Single site for Ce^{3+}: Low disorder, and hence fewer non-radiative pathways.

Ce^{3+} substitutes smaller Y^{3+}: larger 5*d* crystal-field splitting on Ce^{3+}.

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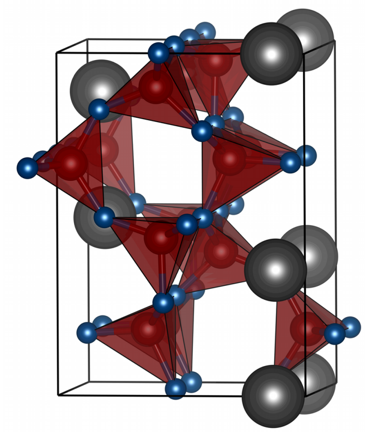
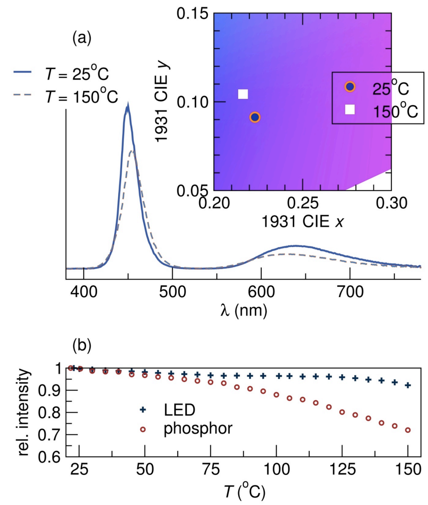
What have we learned? The example of $Sr_2Si_5N_8$

Fully 3-connected tetrahedra, implies stiff lattice.
At the heart of the great thermal stability?





Brinkley, Pfaff, Denault, Hinzten, Seshadri, Nakamura, DenBaars, *Appl. Phys. Lett.* **99** (2011) 241106(1–3).

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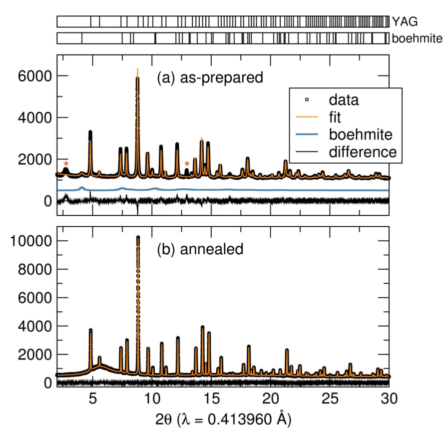
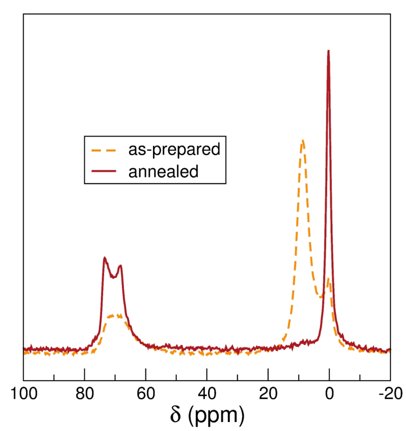
Other research: Efficient nanoscale-YAG:Ce^{3+}

The problem: Solution-prepared YAG:Ce^{3+} nanoparticles (≈30 nm) tend to have significantly suppressed quantum yields. **The solution:** Wrap the nanoparticles in mesoporous SiO_2, anneal, and then dissolve the wrapping.





Synchrotron X-ray (left) and ^{27}Al MAS-NMR (right) of the nanoparticles, before and after annealing.

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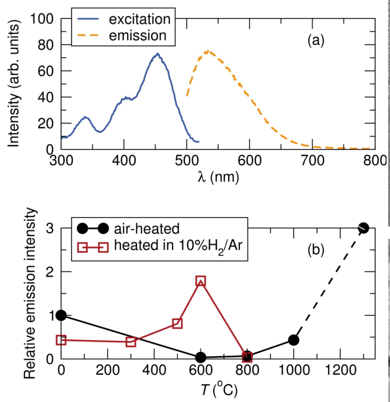
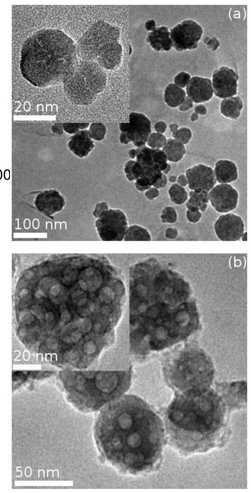
Other research: Efficient nanoscale-YAG:Ce^{3+}

The quantum yield increases from around 30% to around 60% after annealing. The luminescence is stable.



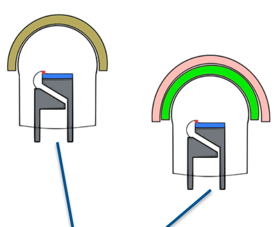


Optical properties and transmission electron microscopy.

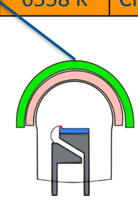
The pores in the final product suggest dehydration.

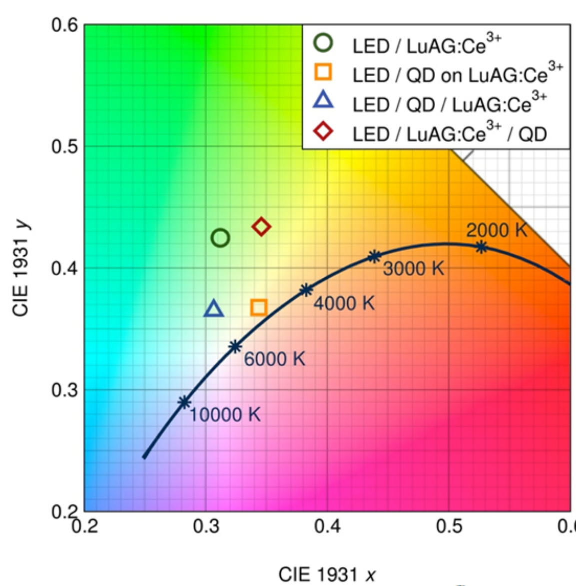
Révaux, Dantelle, George, Seshadri, Gacoin, Boilot, *Nanoscale* **3** (2011) 2015–2022.

Other research: Quantum Dots + garnet phosphors



59 lm/W	5099 K	CRI = 73
96 lm/W	5178 K	CRI = 66
45 lm/W	6558 K	CRI = 71





Denault et al. (unpublished)


Thank you
Photo by Tony Mastres