Ytterbium doped transparent ceramics for solid state laser application

presented by

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Outline

- Background Introduction
  - Solid-state laser, Polycrystalline laser ceramics
- Experimental Process and Results and Analysis
  - Yb:YAG, Yb:LuAG ceramics fabrication and characterization
  - Microstructure, spectroscopic properties and laser experiment
- Summary

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Solid-state laser

Solid state laser

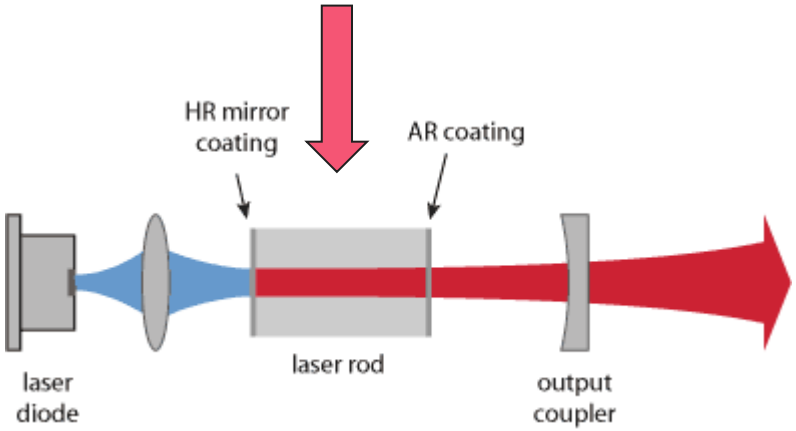
Wide applications in

- Communication
- Material processing
- Medical
- Military etc.

- First working laser invented by Dr. Maiman in 1960s

Ruby single crystal aluminium oxide with chromium (Al_2O_3:Cr)

Solid-state laser



A diode pumped solid state laser setup

Laser gain medium

- Single crystal (monocrystalline material) doped with rare earth (RE) ions or transition metal ions
- E.g.: Neodymium doped Yttrium Aluminium Garnet (Nd:YAG)

- High optical quality
- Good thermal/mechanical properties

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Single crystal laser gain medium

Nd:YAG single crystal boule grown from the melt

YAG Properties	value
Chemical formula	$Y_3Al_5O_{12}$
Molecular weight	593.62
Density	4.55 g/cm^3
Crystal system	cubic
Refractive index	1.81 (1.06μm)
Theoretical transmittance	84.8%
Mohs scale hardness	8~8.5
Melting point	1950˚C
Thermal conductivity	~10 W/m/K

Traditional single crystal

- Grown by Czochralski process
- Very high growing temperature ~ 1950˚C
- Expensive production cost (e.g. Iridium crucible)
- Slow growth rate
- Limited size
- Limited doping concentration (high segregation coefficient)

Researchers has turned refocus on **polycrystalline transparent ceramics** as laser gain medium

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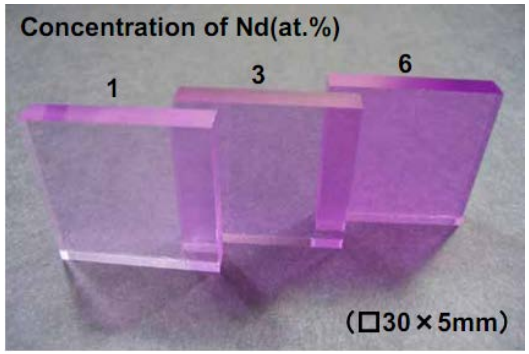
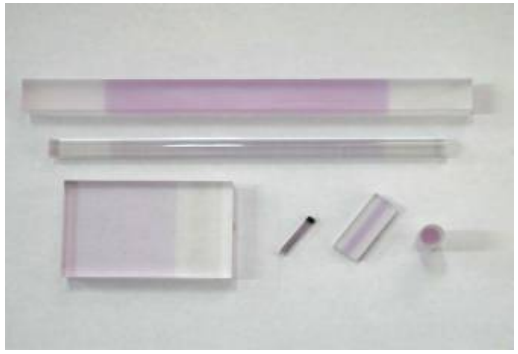
Polycrystalline laser ceramics





Yttrium Aluminium Garnet (YAG) based laser ceramics

Polycrystalline laser ceramics

- Rare earth (RE) element doping: Nd, **Yb**, Ce, Er, Ho, Tm and etc.
- Relatively shorter fabrication period
- High doping concentration
- Large size and composite structures
- Relatively low Sintering temperature (~70% of melting temperature)
- Cheaper production cost
- Optical/thermal/mechanical properties comparable with single crystal

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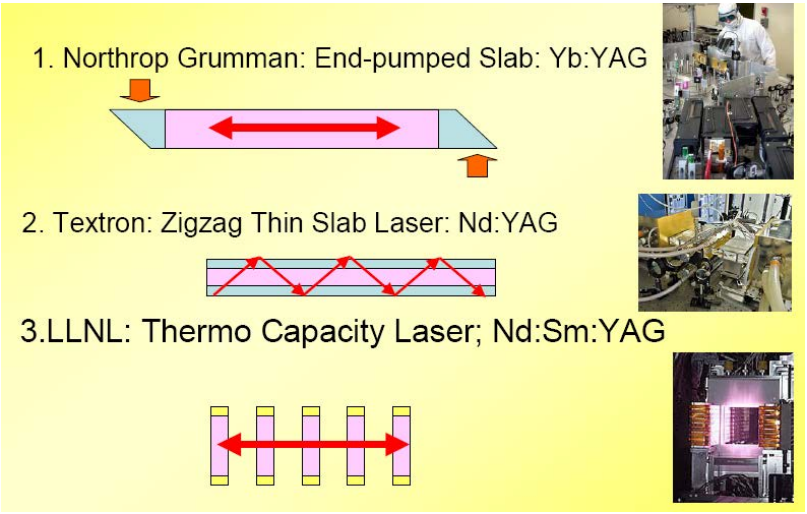
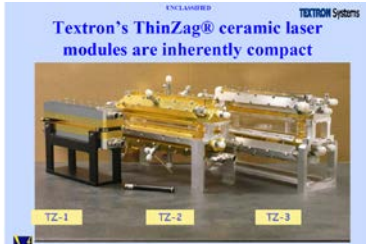
Development in laser ceramics

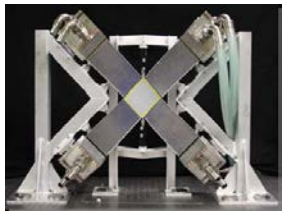
- **Development of laser ceramics**
  - 1990: Haneda et al. reported transparent Nd:YAG ceramics obtained by a urea precipitation method, with nearly same optical properties as those of single crystal.
  - 1995: Ikesue et al. fabricated high quality Nd:YAG ceramics by reactive sintering method. And achieved **first** laser operation with 70 mW laser power
  - 2000: Yanagitani et al. reported Nd:YAG ceramics (prepared by wet chemical method) of 357 mW and 53% slope efficiency, close to Nd:YAG single crystal (54.5%)
  - 2001: 1.46 kW Nd:YAG ceramics laser demonstrated

Development in laser ceramics

- **Development of laser ceramics**







  - 2006: LLNL's solid-state heat capacity laser: 67 kW output power
  - 2009: Northrop Grumman:105 kW ceramics laser

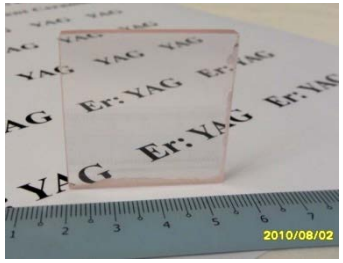
The advanced modern ceramics processing technology greatly pushed the development of ceramics lasers,

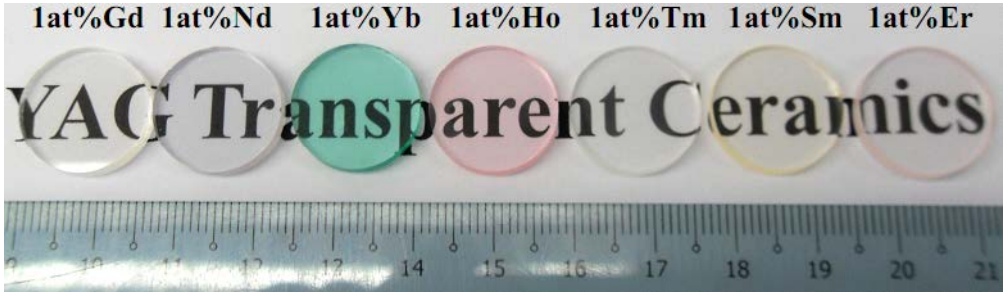
yet it's still a challenging work to obtain high quality laser ceramics

Research of laser ceramics in NTU

Mainly on RE:YAG laser ceramics

– Rare earth (RE) element doping: Nd, **Yb**, Er, Ho, Tm and etc.





We have gradually formed up the fabrication capability.

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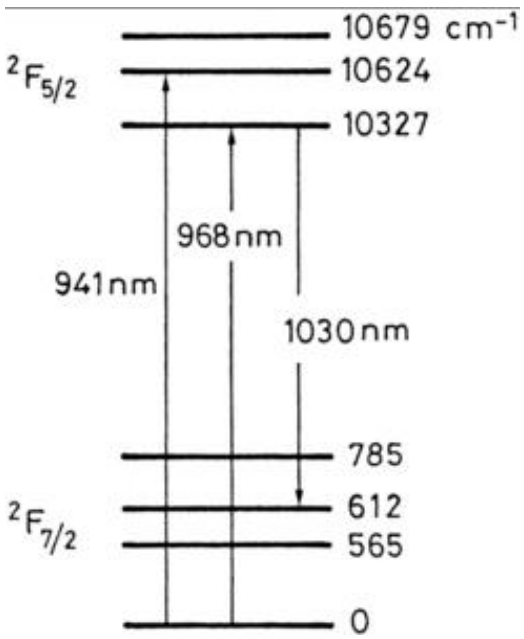
Trivalent Ytterbium (Yb^{3+}) ions



Energy levels of Yb^{3+} ions in Yb:YAG

- **Yb^{3+} ion energy structure**
  - Quasi-three-level system
  - Simple electronic level structure with two manifolds
  - No upconversion/ No excited state absorption/ No cross relaxation/ No concentration quenching
  - Low quantum defect (~9%)
  - High efficiency
  - Broadband suitable for direct InGaAs laser diode pumping
  - Long fluorescent lifetime

Suitable for high power CW laser and ultra-short pulse laser application

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Objective

- **Objective of the research**
  - Establish and optimize the fabrication method of the Ytterbium-doped laser ceramics
    - **Yb:YAG** ceramics and **Yb:LuAG** ceramics,
  - Study on the microstructure properties
  - Investigation on the spectroscopic properties
    - Optical transmittance, absorption/emission spectra
  - Characterization of the laser performance
    - Continuous Wave (CW) laser operation
  - Development of new laser ceramics

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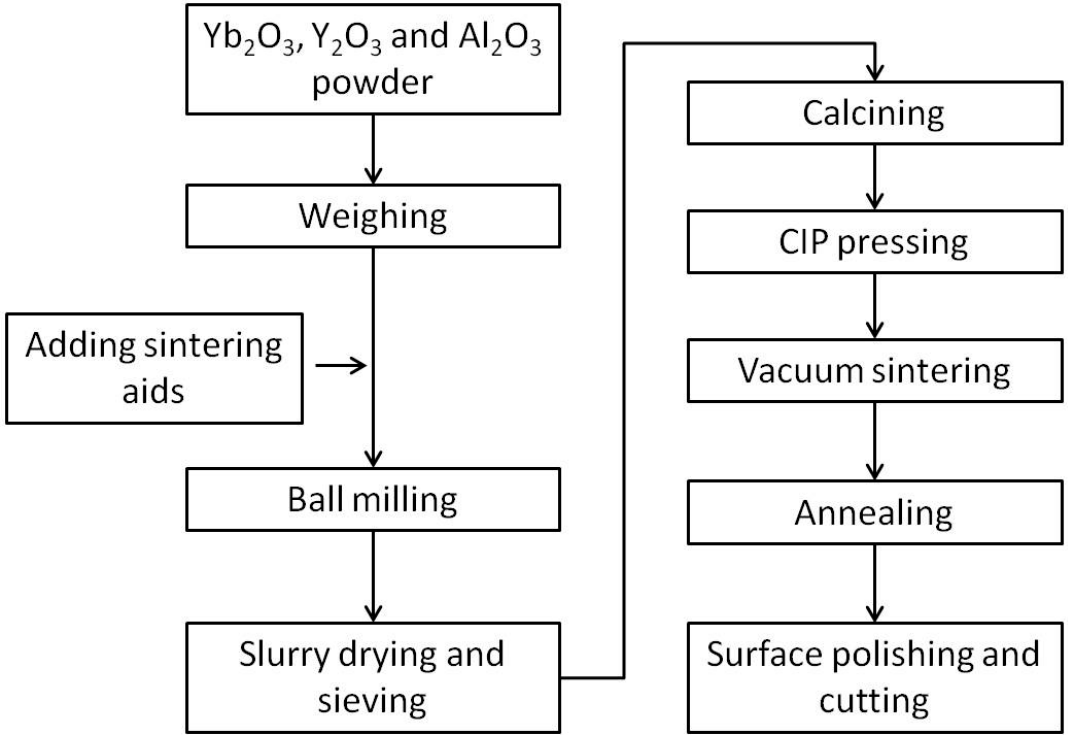
Yb:YAG ceramics fabrication



Flowchart for fabricating the Yb:YAG laser ceramics

Yb:YAG laser ceramics

- In-house fabrication
- Reactive sintering method
- Short fabrication period: around 1 week

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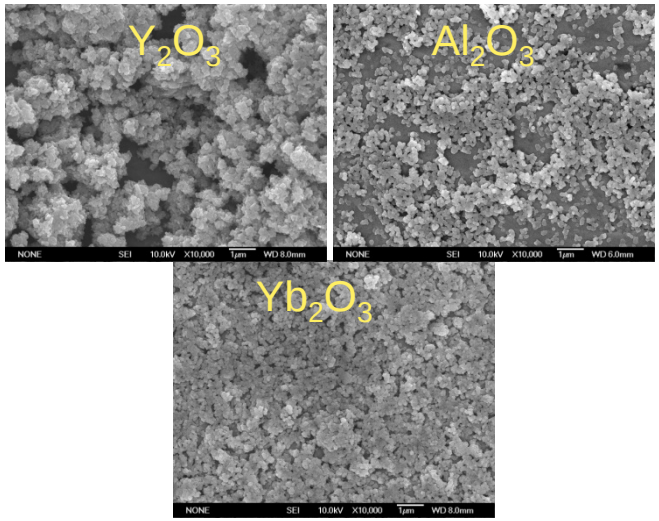
Yb:YAG ceramics fabrication

Raw Material: High purity Al_2O_3, Y_2O_3 and Yb_2O_3 powder (sub-micron size)

Weighed precisely according to chemical stoichiometric composition 1at.%~20at.% Yb:YAG

Ball milled with ethanol. Tetraethyl orthosilicate (TEOS) added to introduce SiO_2



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Yb:YAG ceramics fabrication

Dry pressed and further cold isostatic pressed (CIP) at 200MPa to form green body

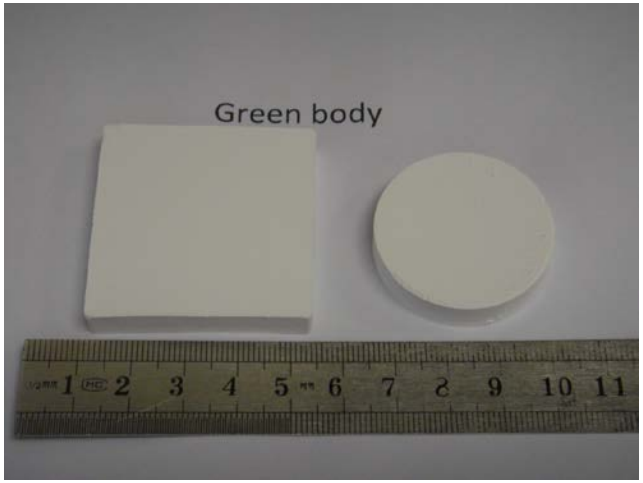
→

Sintered using a high temperature vacuum sintering furnace

→

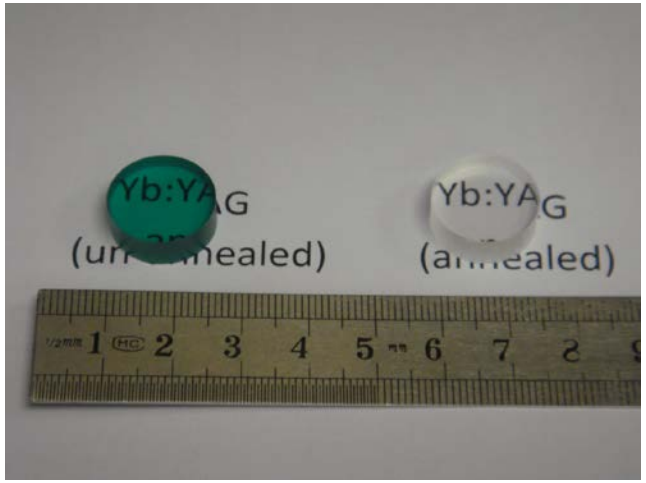
Grinded and mirror polished for surface flatness

Yb:YAG ceramic samples



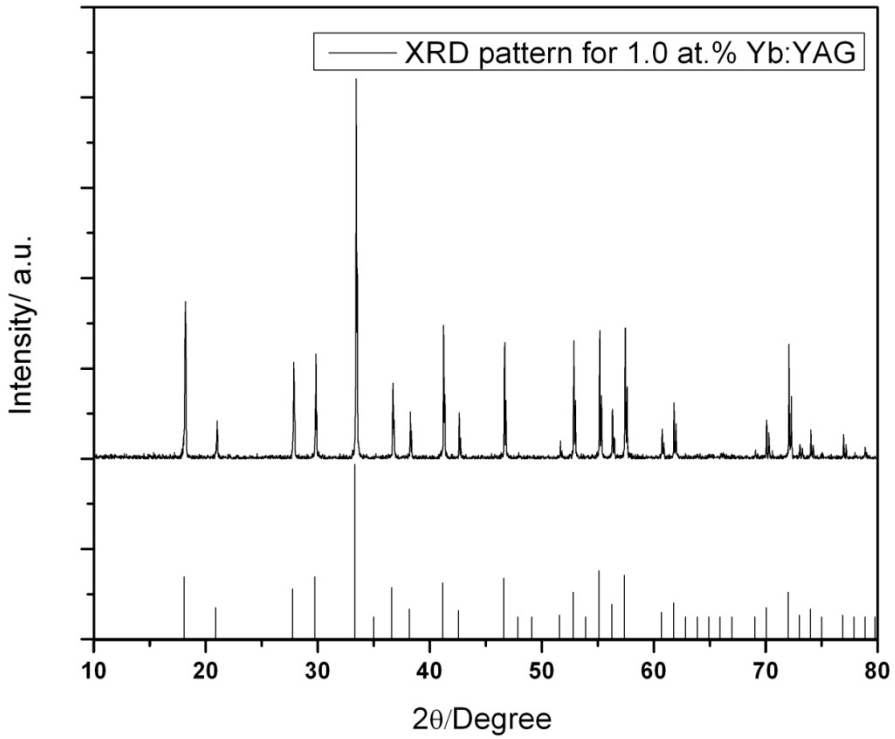
Green-body

Sintered samples



Polished samples

XRD Results



XRD pattern of the 1.0at.% Yb:YAG ceramics sintered at 1770^0C for 8h

Yb:YAG laser ceramics

- XRD pattern shows YAG phase
- No other phase is present

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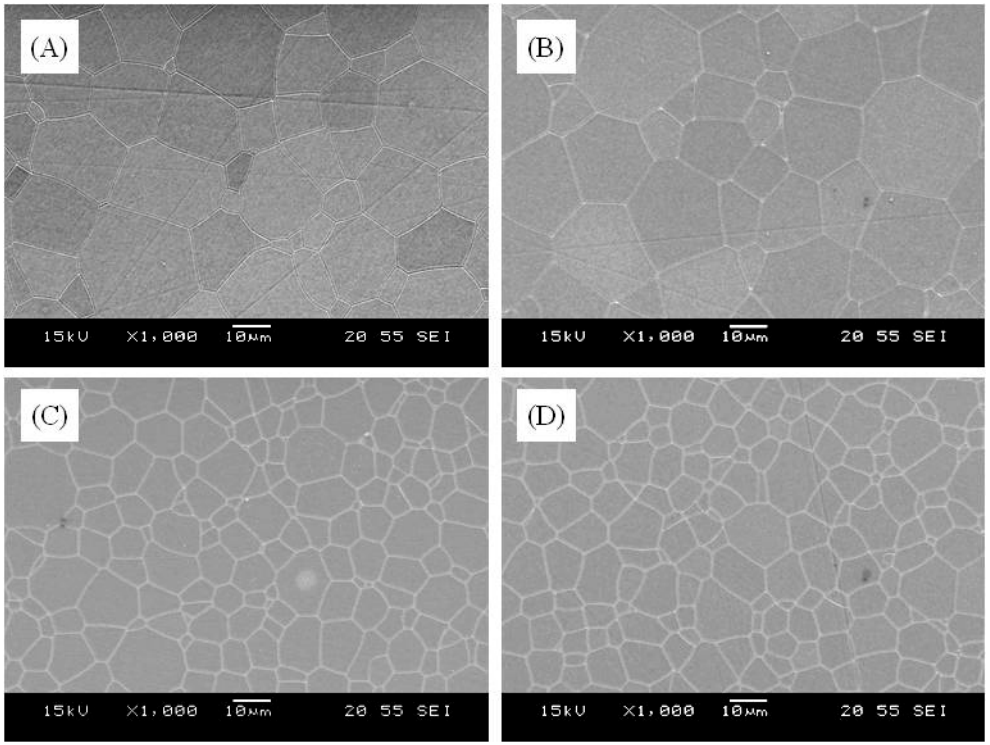
SEM images



Surface SEM images of the Yb:YAG ceramics:
(A)1at.%, (B)8at.%, (C) 15at.% and (D)20at.%

Yb:YAG laser ceramics

- Dense and almost pore free microstructure
- Grain size decrease slightly with higher Yb^{3+} doping concentration
- Average grain size around 10 μm

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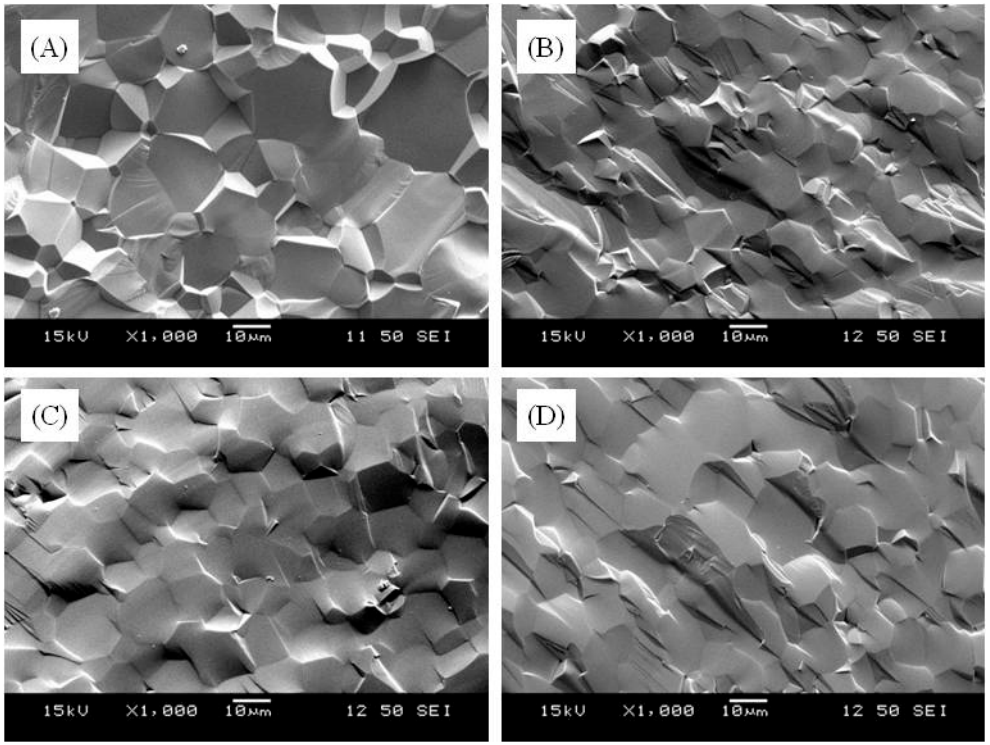
SEM images



Fracture SEM images of the Yb:YAG ceramics:
(A)1at.%, (B)8at.%, (C) 15at.% and (D)20at.%

Yb:YAG laser ceramics

- Dense and almost pore free microstructure
- Grain size decrease slightly with higher Yb^{3+} doping concentration
- Average grain size around 10 μm

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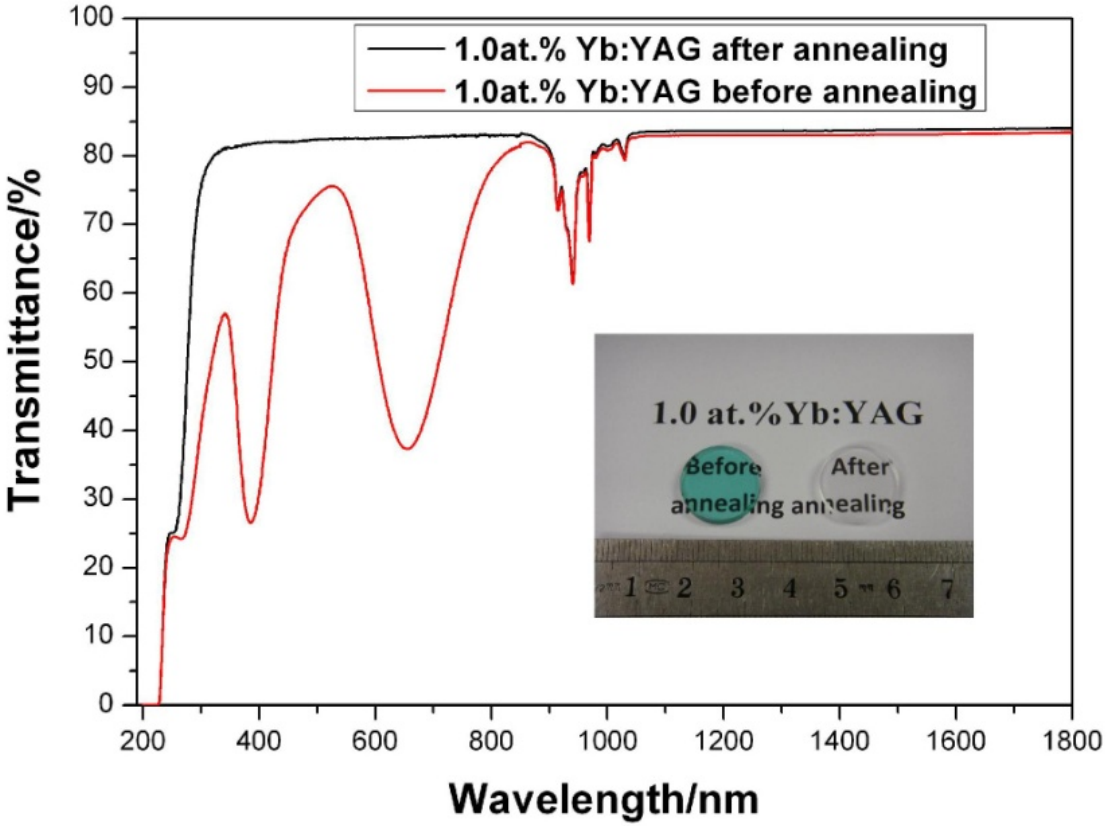
Optical transmittance



Transmittance curve of 1.0 at.% Yb:YAG ceramics (3 mm thickness) before and after annealing process

Yb:YAG laser ceramics

- Transparent after polishing
- Samples are green in color before annealing and appear colorless after annealing
- Un-annealed sample: two absorption bands at 375 nm and 625 nm
- Due to presence of Yb^{2+} and Re-F color center
- Transmittance value reach 83% at 1300 nm and remain 81% at 400 nm

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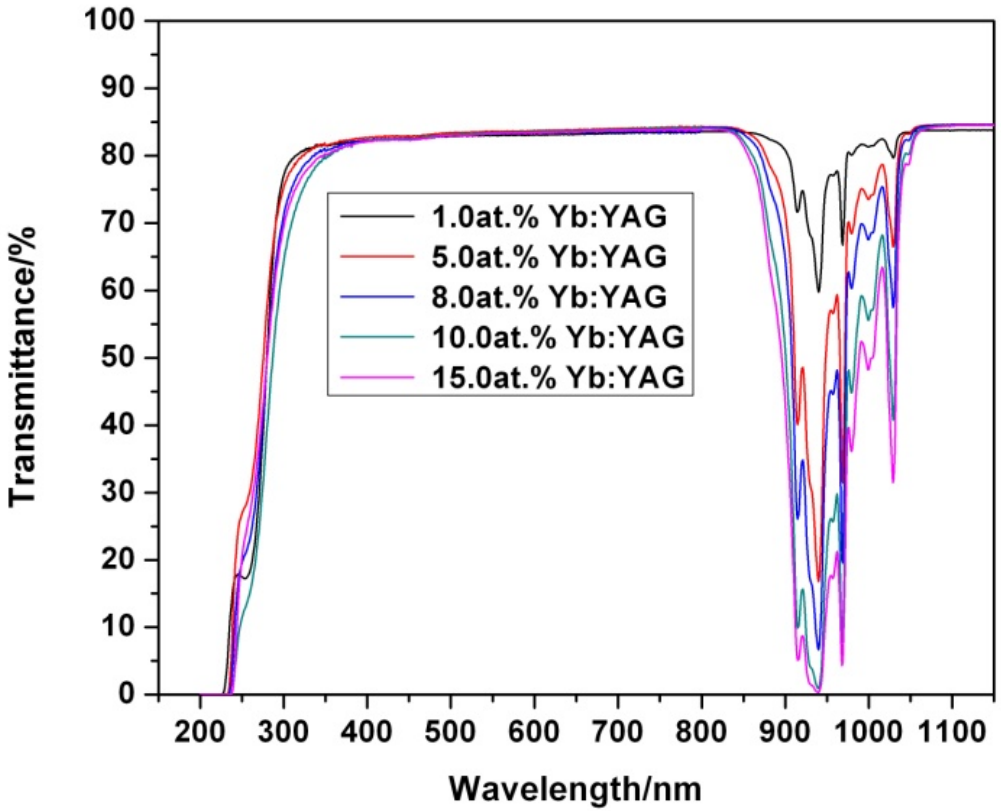
Optical transmittance



Transmittance curves of the Yb:YAG ceramic samples (~3 mm thickness) with different doping concentrations

Yb:YAG laser ceramics

- All samples shows high optical transmittance
- Absorption band from 800 nm to 1050 nm
- Very low transmittance value for samples of high doping at absorption peak

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Absorption coefficient calculation

Optical Transmittance:

$$T=\frac{I}{I_0}=\frac{(1-R)^2}{1-R^2e^{-2\alpha t}}e^{-2\alpha t}$$

Theoretical transmittance:

$$T=\frac{I}{I_0}=\frac{(1-R)^2}{1-R^2}=\frac{2n}{n^2+1}$$

$$R=\frac{(n-1)^2}{(n+1)^2}$$

where R is surface reflectance, α is absorption coefficient of Yb:YAG, and n is refractive index of material,
Thus, the absorption coefficient α_{abs} of Yb:YAG could be calculated by:

$$\alpha_{abs}=\frac{1}{t}\ln\left(\frac{(1-R)^2}{2T}+\sqrt{R^2+\frac{(1-R)^2}{4T^2}}\right)$$

Where t is the sample thickness

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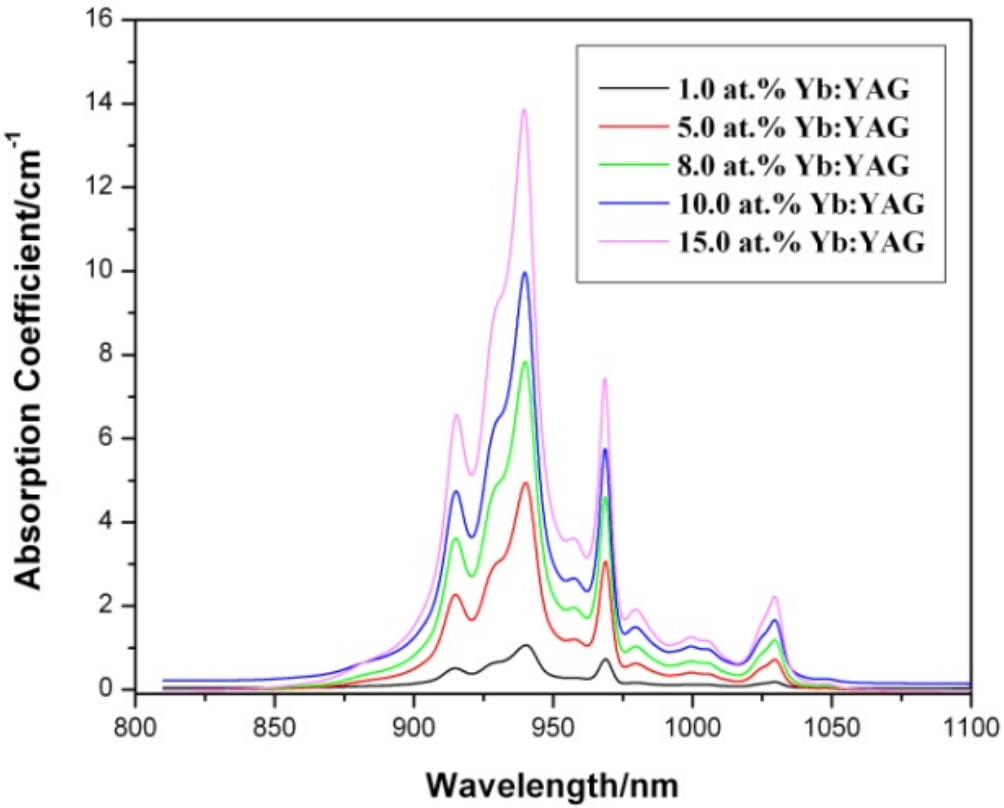
Absorption spectra



Absorption spectra of the Yb:YAG ceramic samples with different doping concentrations

Yb:YAG laser ceramics

- Absorption peak at 940 nm
- Absorption coefficient increases with increase of Yb^{3+} doping
- Two moderate absorption band at 915 nm and 969 nm
- Re-absorption at 1030 nm
- Broad absorption band suitable for direct InGaAs laser diode pumping

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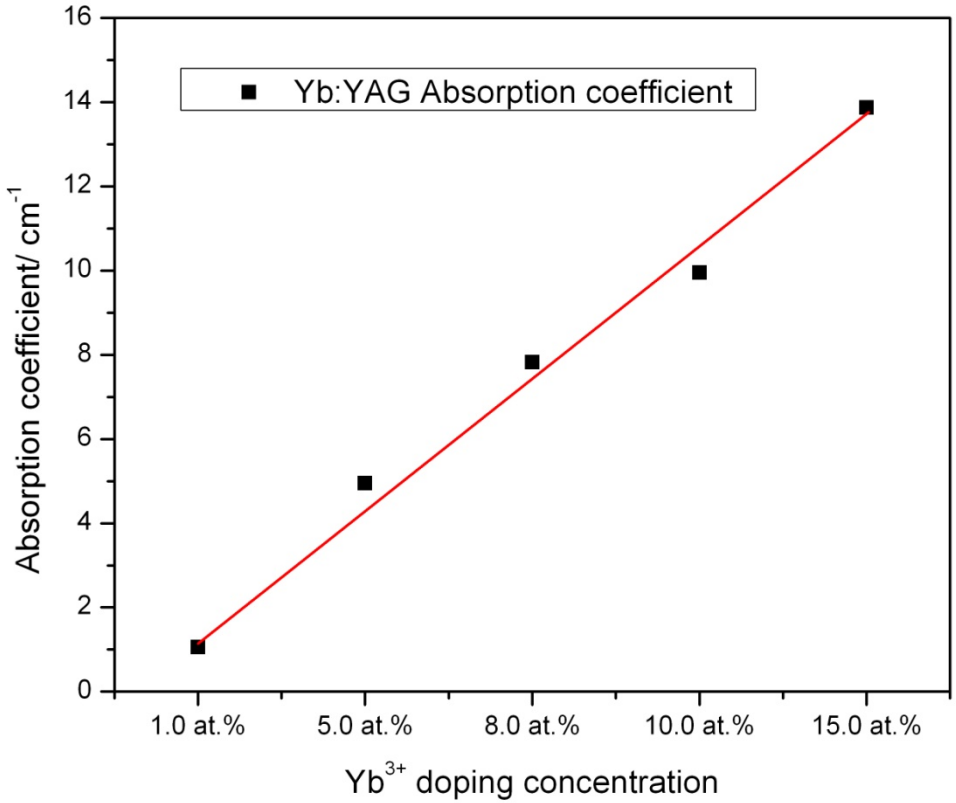
Absorption spectra



Absorption spectra of the Yb:YAG ceramic samples with different doping concentrations

Yb:YAG laser ceramics

- Absorption peak at 940 nm
- Absorption coefficient:

Yb^{3+} doping concentration	Absorption coefficient @940nm (cm^{-1})
1.0 at.%	1.06
5.0 at.%	4.95
8.0 at.%	7.83
10.0 at.%	9.95
15.0 at.%	13.87

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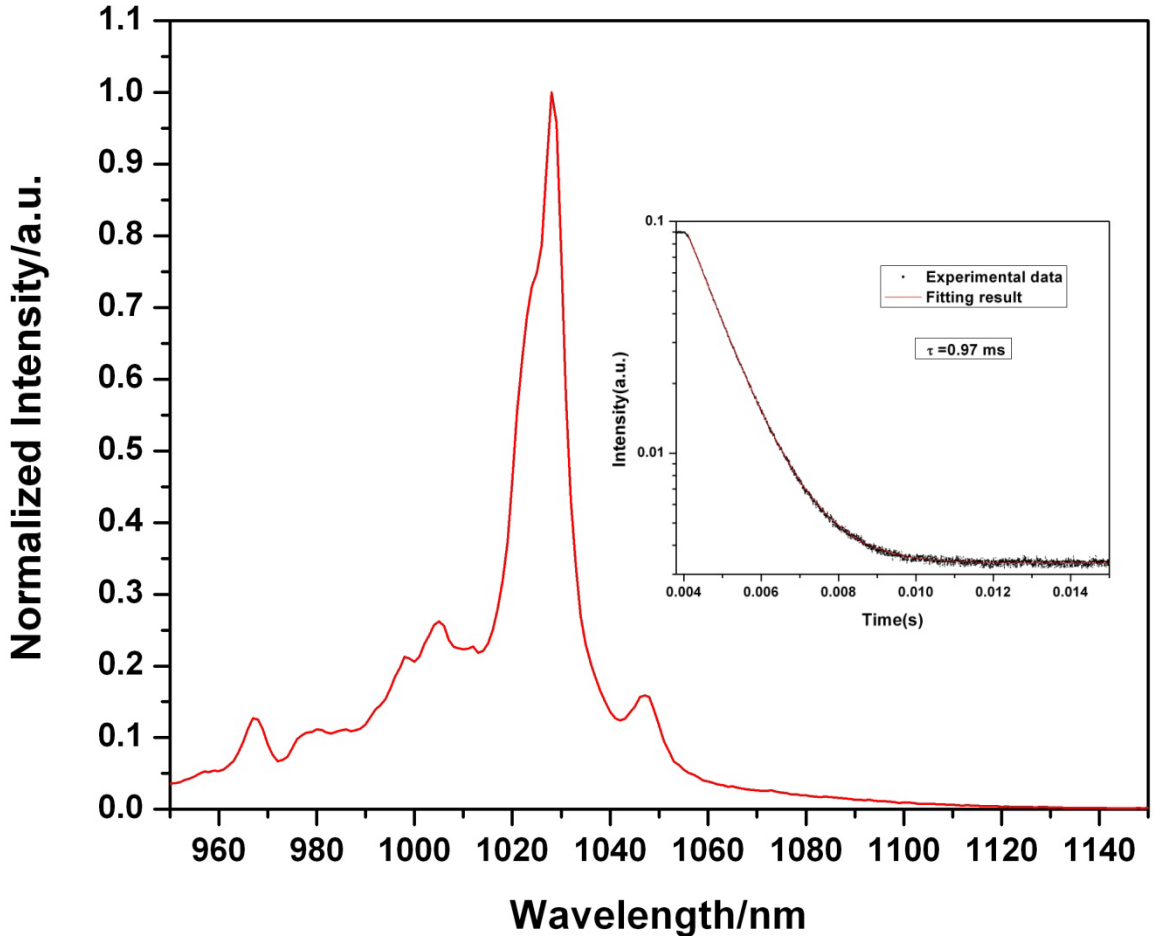
Emission spectrum



The emission spectrum of fabricated 5.0 at.% Yb:YAG ceramic sample

Yb:YAG laser ceramics

- Emission peak at 1028 nm
- Full width half maximum (FWHM) measured to be 11.07 nm
- Broad emission band is useful in generating ultra-short pulse in mode-locked lasers
- Fluorescent lifetime

  5% Yb:YAG: 0.97 ms

  10% Yb:YAG: 0.95 ms

  15% Yb:YAG: 0.95 ms

  (no concentration quenching)

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Absorption/Emission cross-section

– The absorption cross-section is calculated by:

$$\sigma_{abs}(\lambda) = \frac{\alpha_{abs}(\lambda)M}{\rho N_A C}$$

Where α_{abs} is absorption coefficient of Yb:YAG, M is the molar mass, ρ is the density and C is the Yb doping concentration.

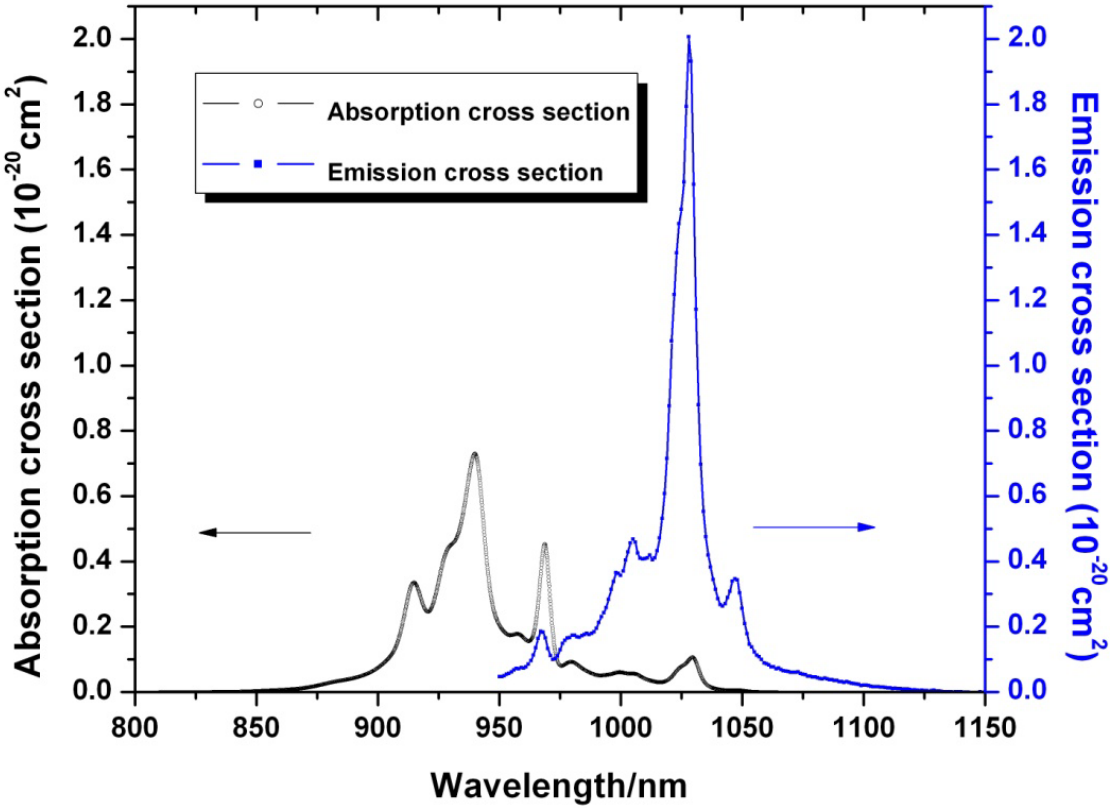
– The emission cross-section is calculated by Fuchtbauer-Landenburg (F-L formula)*:

$$\sigma_{em}(\lambda) = \frac{\lambda^5 I(\lambda)}{8\pi n^2 c\tau_r \int I(\lambda)\lambda d\lambda}$$

Where I(λ) is the intensity of emission spectrum, and τ is the fluorescent lifetime.

* B. F. Aull, and H. P. Jenssen, IEEE J. Quantum Electron. QE-18, 925 (1982)

Absorption/Emission cross-section



Absorption and emission cross-section of fabricated Yb:YAG ceramic sample

Yb:YAG laser ceramics

- Absorption bandwidth: 19.5 nm
- Emission bandwidth: 11.07 nm
- Absorption cross-section at 940 nm: 0.72×10^{-20}cm^2
- Emission cross-section at 1028 nm: 2.01×10^{-20}cm^2
- Comparable with Yb:YAG single crystal (2.3×10^{-20} cm^2) reported*.

*D. S. Sumida et al. , Optics Letters, Vol. 19, Issue 17, pp. 1343-1345 (1994)

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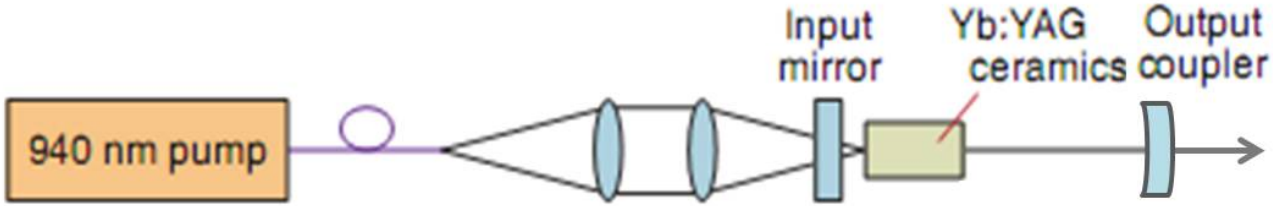
Continuous wave laser performance

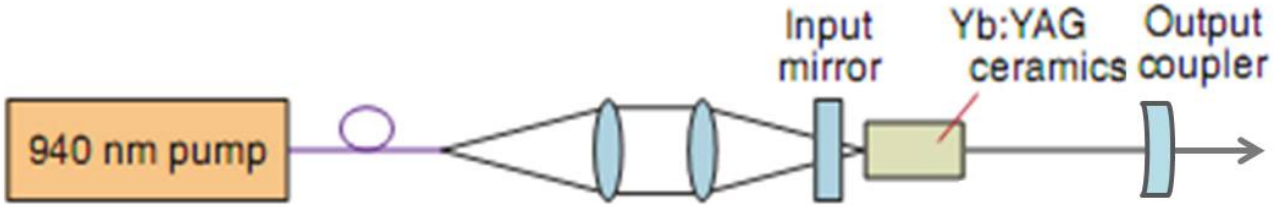
- **End-pumped two mirror cavity setup**



  - Pump source: Limo025-F100-DL940, fiber-coupled
  - Fiber core size: 100μm
  - Sample size: 3*3*3mm, AR coated @ 940 nm and 1030 nm
  - Cavity length: ~30mm
  - Input mirror: HR coated @ 940 nm and 1030 nm
  - Output coupler: Toc= 10%
  - Cooling temperature: around 17℃

Continuous wave laser performance

- **End-pumped two mirror cavity setup**



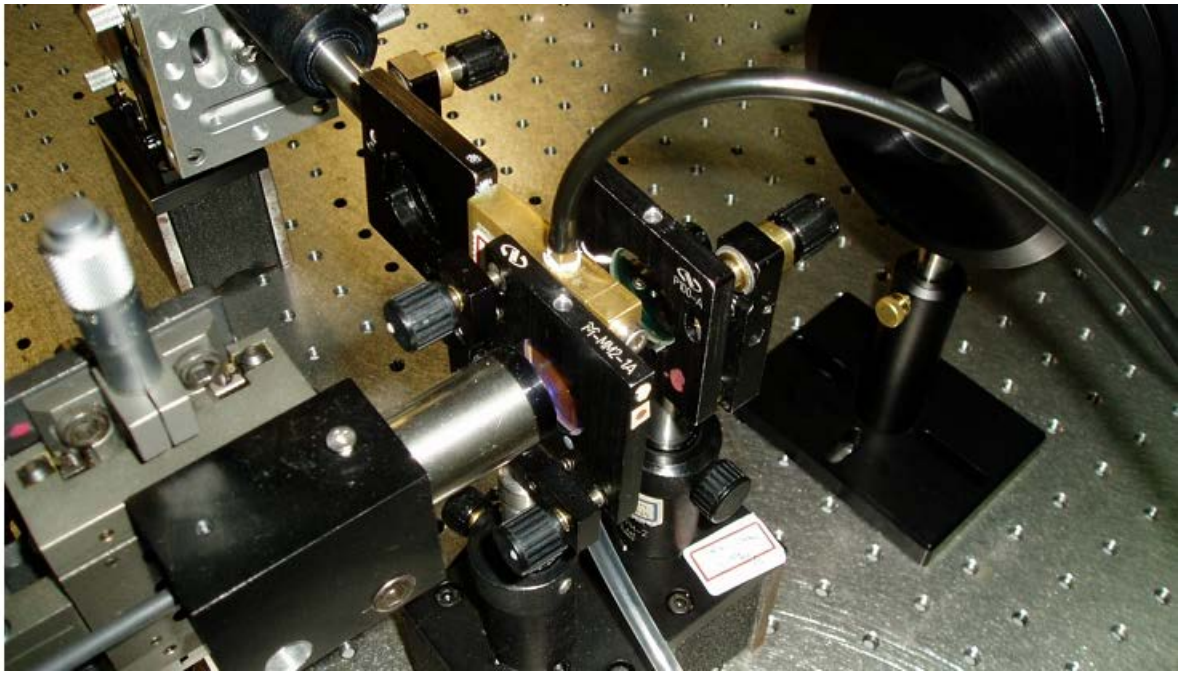
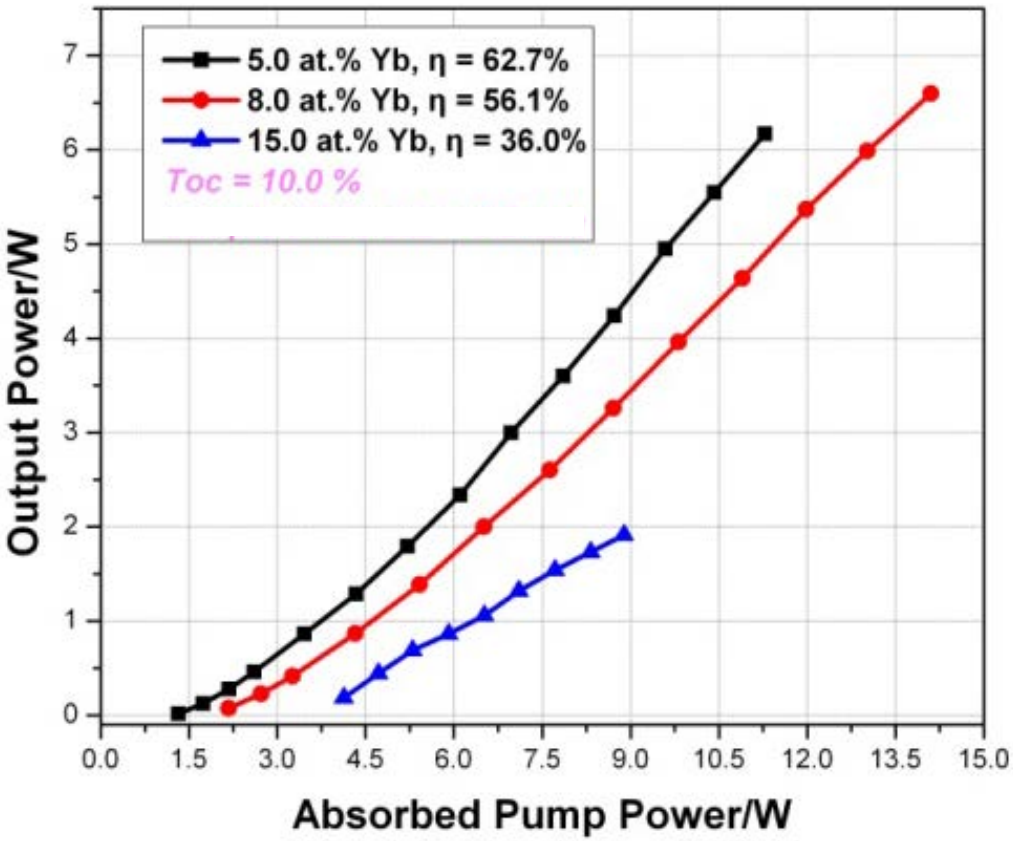


Photo of the CW laser setup

Continuous wave laser performance



Laser performance of Yb:YAG ceramic samples

Yb:YAG laser ceramics

- CW Laser operation was achieved
- Maximum laser output power of 6.9 W obtained by a 8.0 at.% Yb:YAG
- Slope efficiency of 62.7% obtained with a 5.0 at.% Yb:YAG
- Optimization of laser setup is still possible

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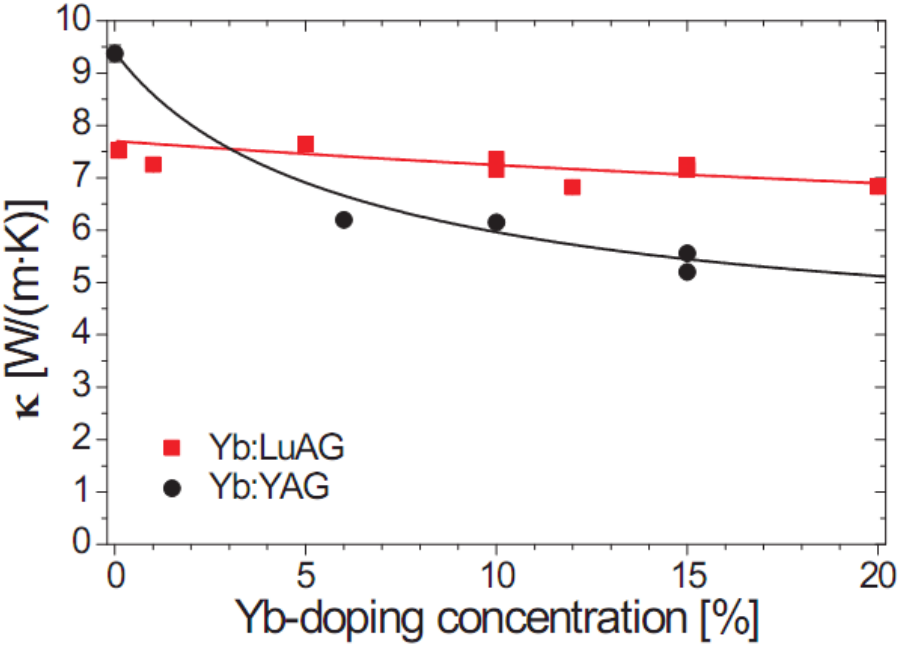
Yb doped Lutetium Aluminium Garnet (Yb:LuAG) laser ceramics



Thermal conductivity of Yb:YAG and Yb:LuAG with different Yb doping *

* Beil et al. Optics Express, Vol. 18, Issue 20, pp. 20712-20722 (2010)

Yb:LuAG laser ceramics

- Yb:$Lu_3Al_5O_{12}$
- Small mass difference between Yb and Lu
- Low phonon scattering
- Small drop of thermal conductivity in high Yb doping
- Suitable for high power thin disk laser application

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Yb:LuAG ceramics fabrication

Yb:LuAG laser ceramics

- Raw materials: High purity Yb_2O_3, Lu_2O_3 and Al_2O_3 powders
- Fabrication process similar to that of Yb:YAG ceramics

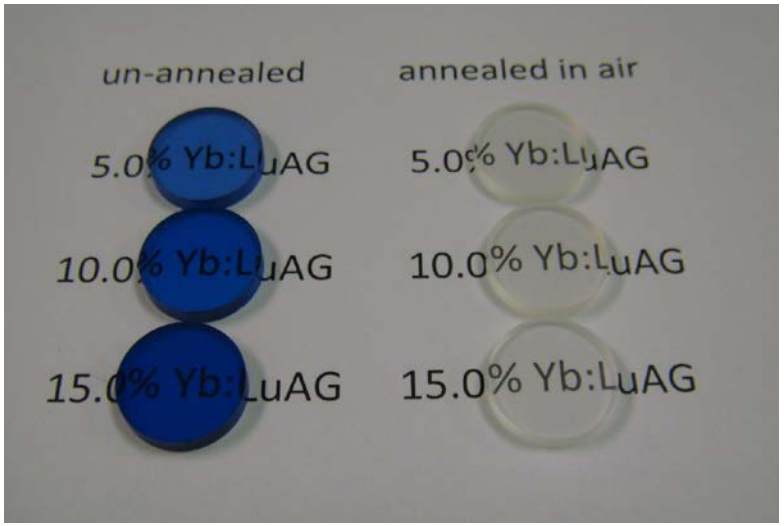

Photo of the Yb:LuAG ceramic samples sintered at 1840˚C with different doping concentrations

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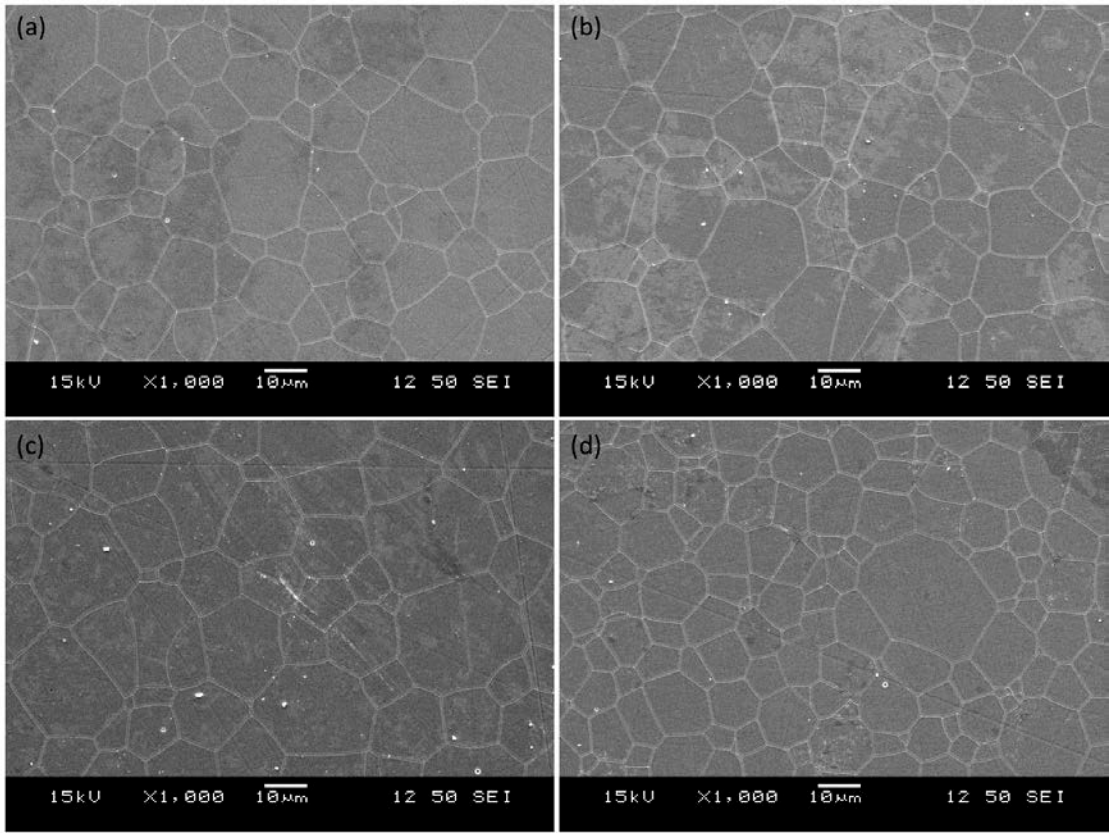
SEM images of Yb:LuAG ceramics



Yb:LuAG laser ceramics

- Minor pores can be found
- Average grain size around 10 μm

Surface SEM images of the Yb:LuAG ceramics: (a)5at.%, (b)10at.%, (c) 15at.% and (d)20at.% sintered at 1840^0C for 8h

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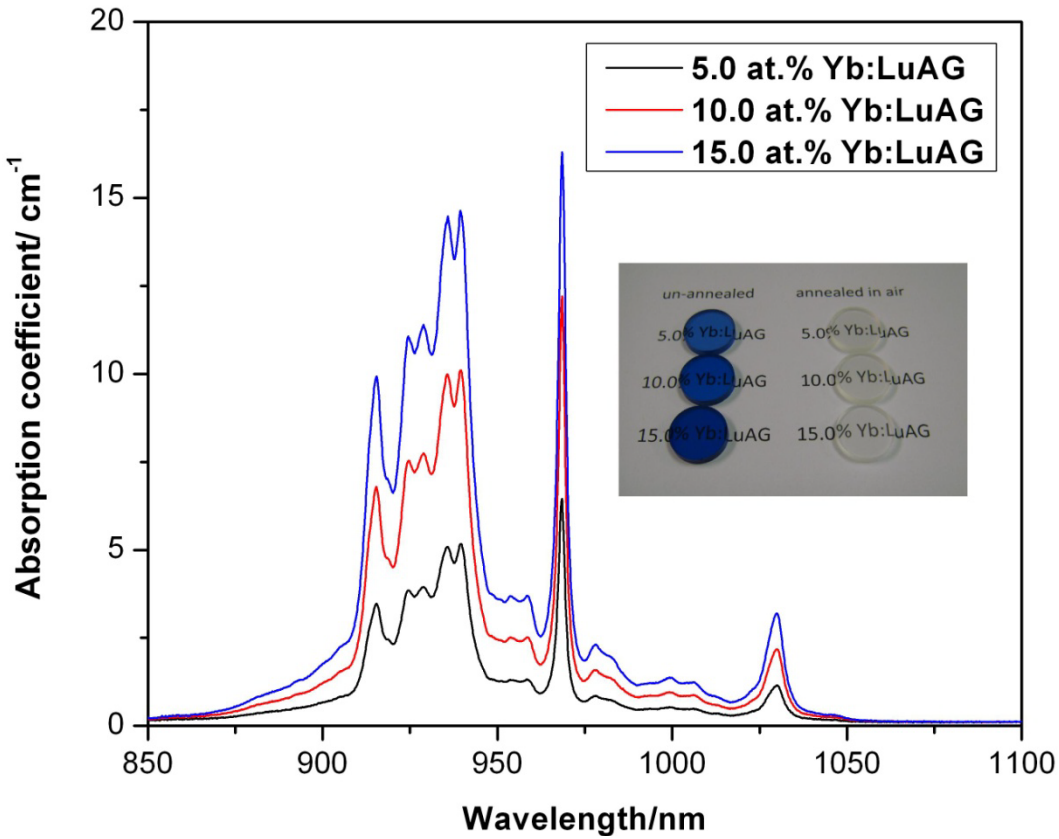
Absorption spectra of Yb:LuAG ceramics



Absorption spectra of the Yb:LuAG ceramic samples with different doping concentrations

Yb:LuAG laser ceramics

– Absorption coefficient:

Yb^{3+} doping concentration	Absorption coefficient @968nm (cm^{-1})
5.0 at.%	6.4
10.0 at.%	12.2
15.0 at.%	16.3

– Broad absorption band at 940 nm with double peaks centered at 935.6 nm and 939.6 nm

– FWHM: 21.6 nm

– Absorption cross-section at 939 nm: $0.73 \times 10^{-20} cm^2$
968 nm: $0.91 \times 10^{-20} cm^2$

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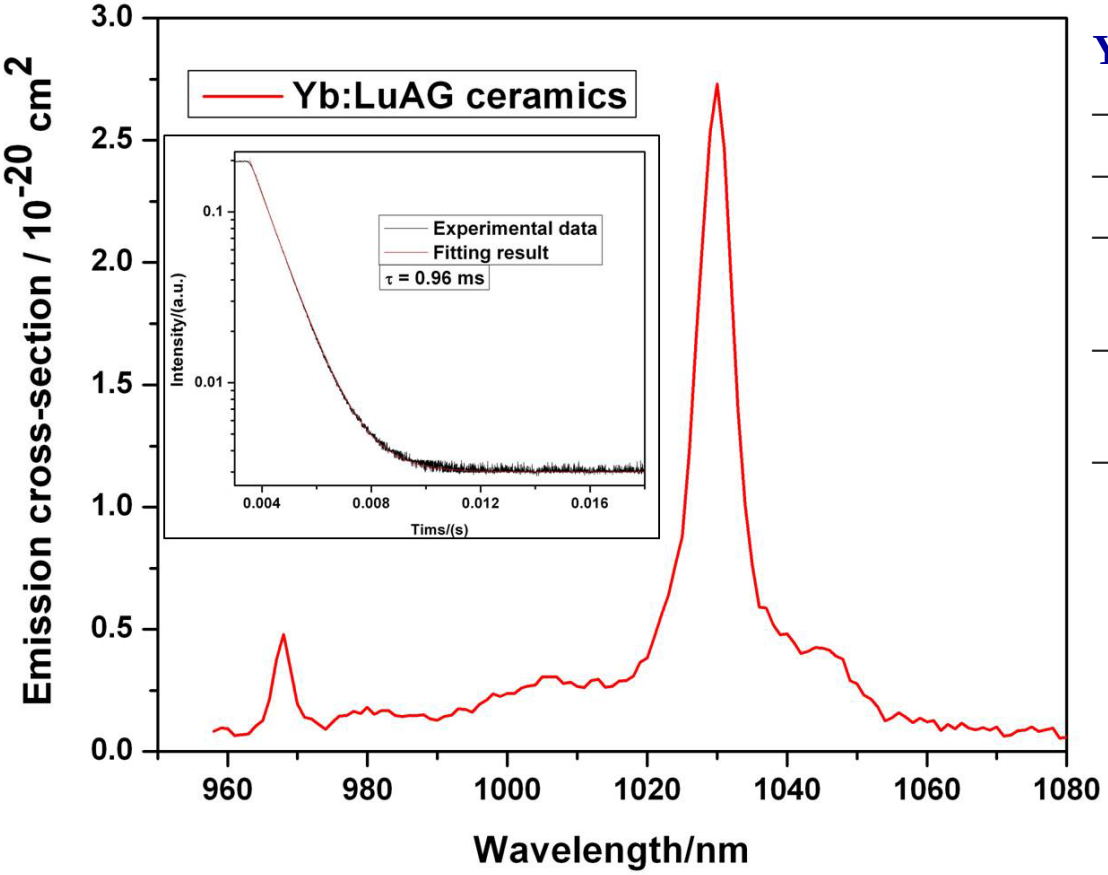
Emission spectrum of Yb:LuAG ceramics



The emission spectrum of fabricated 5.0 at.% Yb:LuAG ceramic sample

Yb:LuAG laser ceramics

- Emission peak at 1030 nm
- FWHM of 6.8 nm
- Fluorescent lifetime was measured to be 0.96 ms
- Emission cross-section at 1030 nm: $2.7 \times 10^{-20} cm^2$
- 30% higher compared to Yb:YAG ($2.01 \times 10^{-20} cm^2$)

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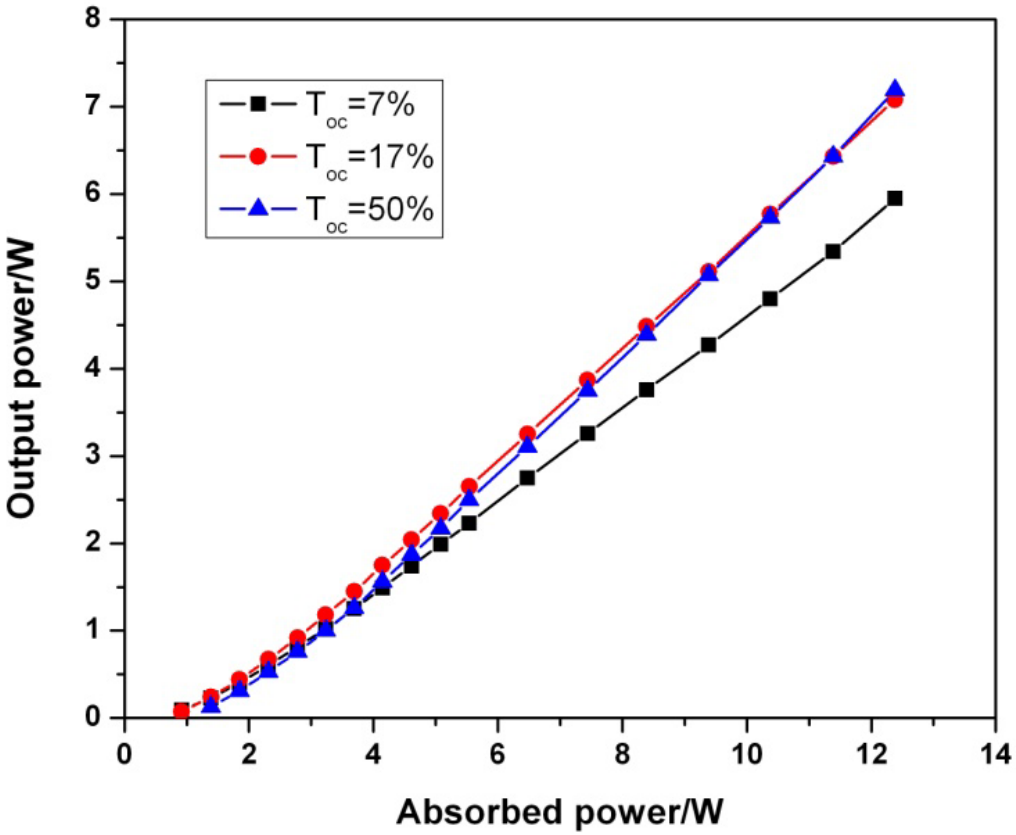
Continuous wave laser performance



Laser performance of 5.0 at.% Yb:LuAG ceramic sample

Yb:LuAG laser ceramics

- First Yb:LuAG ceramics laser reported
- Maximum laser output power of 7.2 W obtained
- Corresponding to a slope efficiency of 65 %

  (72% reported of Yb:LuAG single crystal *)

*J. Dong et al. Laser Physics Letters, Vol. 7, pp7 26–733 (2010)

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Summary

Yb:YAG and Yb:LuAG laser ceramics

- High quality Yb:YAG and Yb:LuAG laser ceramics obtained
- CW laser operation successfully demonstrated

Properties	Yb:YAG ceramics	Yb:LuAG ceramics
Absorption cross-section @ 940 nm	$0.72 \times 10^{-20} cm^2$	$0.73 \times 10^{-20} cm^2$
Absorption bandwidth	19.5 nm	21.6 nm
Emission cross-section @ 1030 nm	$2.01 \times 10^{-20} cm^2$	$2.7 \times 10^{-20} cm^2$
Emission bandwidth	11.0 nm	6.8 nm
Highest Laser slope efficiency	62.7%	65.0%

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Acknowledgement

- The presenter would like to dedicate this talk to :

  Professor Ma jan
  Former Chair of School of Materials Science and Engineering (MSE)
  Nanyang Technological University

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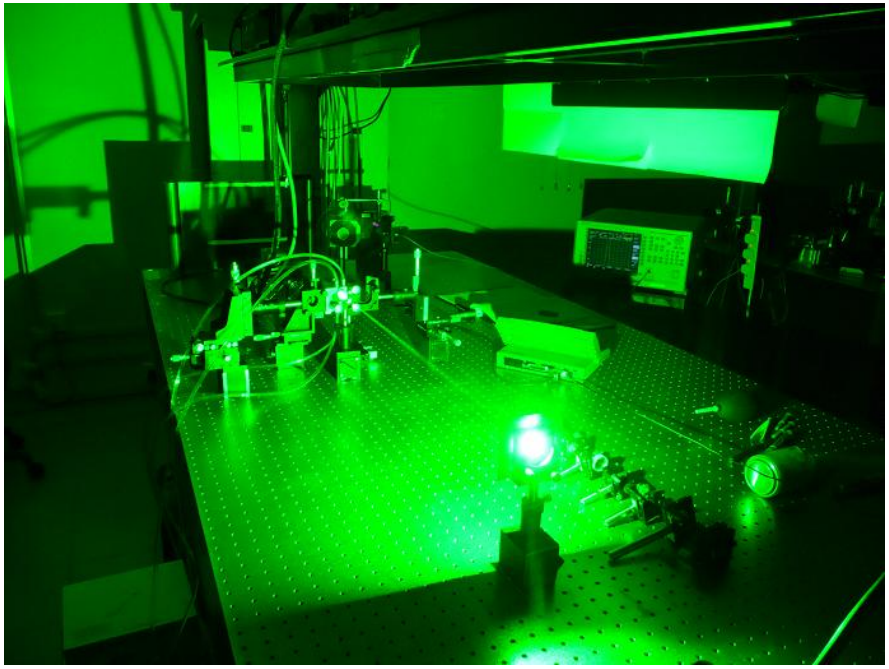
Thank you!



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