Synthesis of nano-sized phosphors in supercritical water conditions

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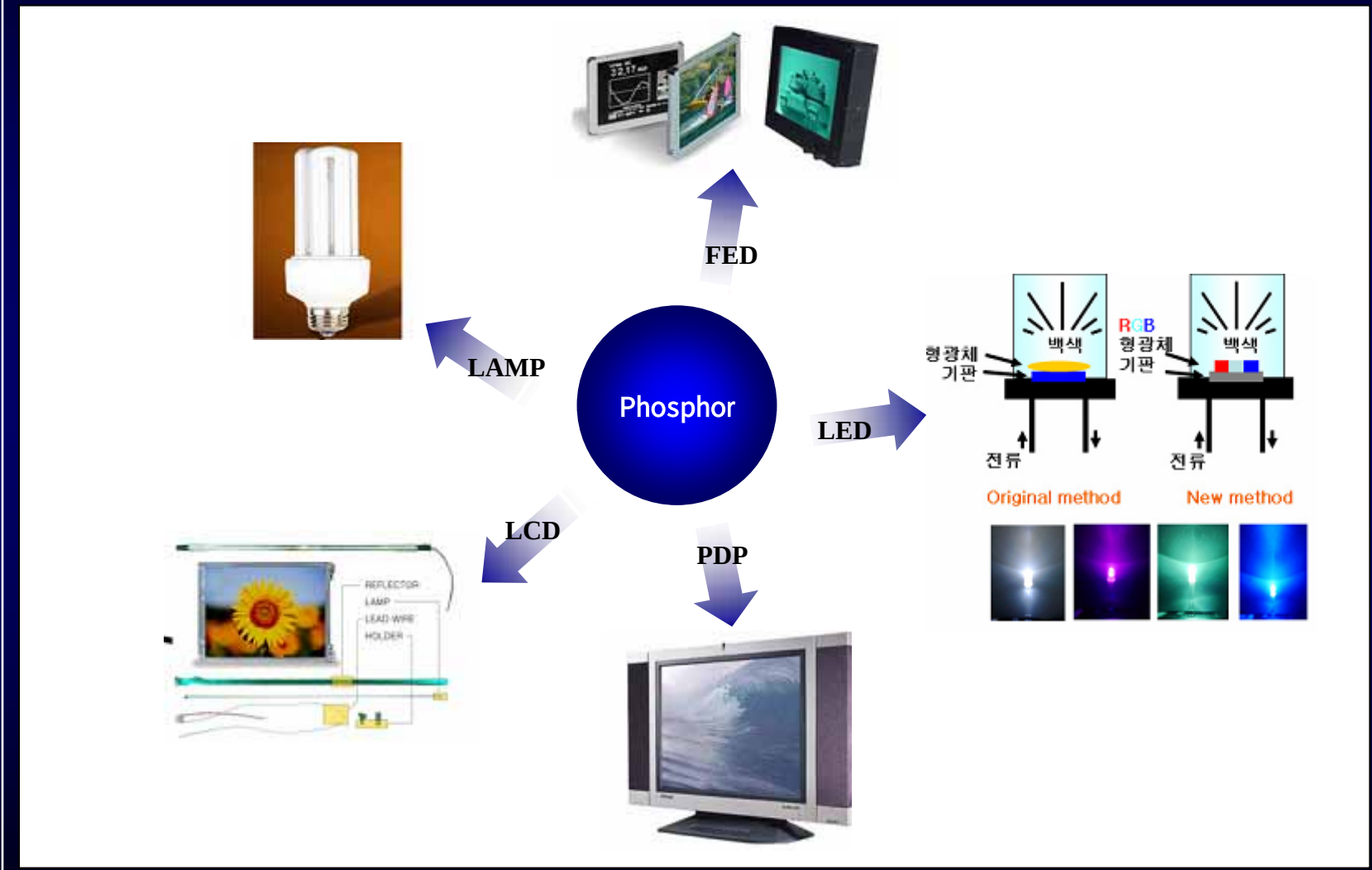
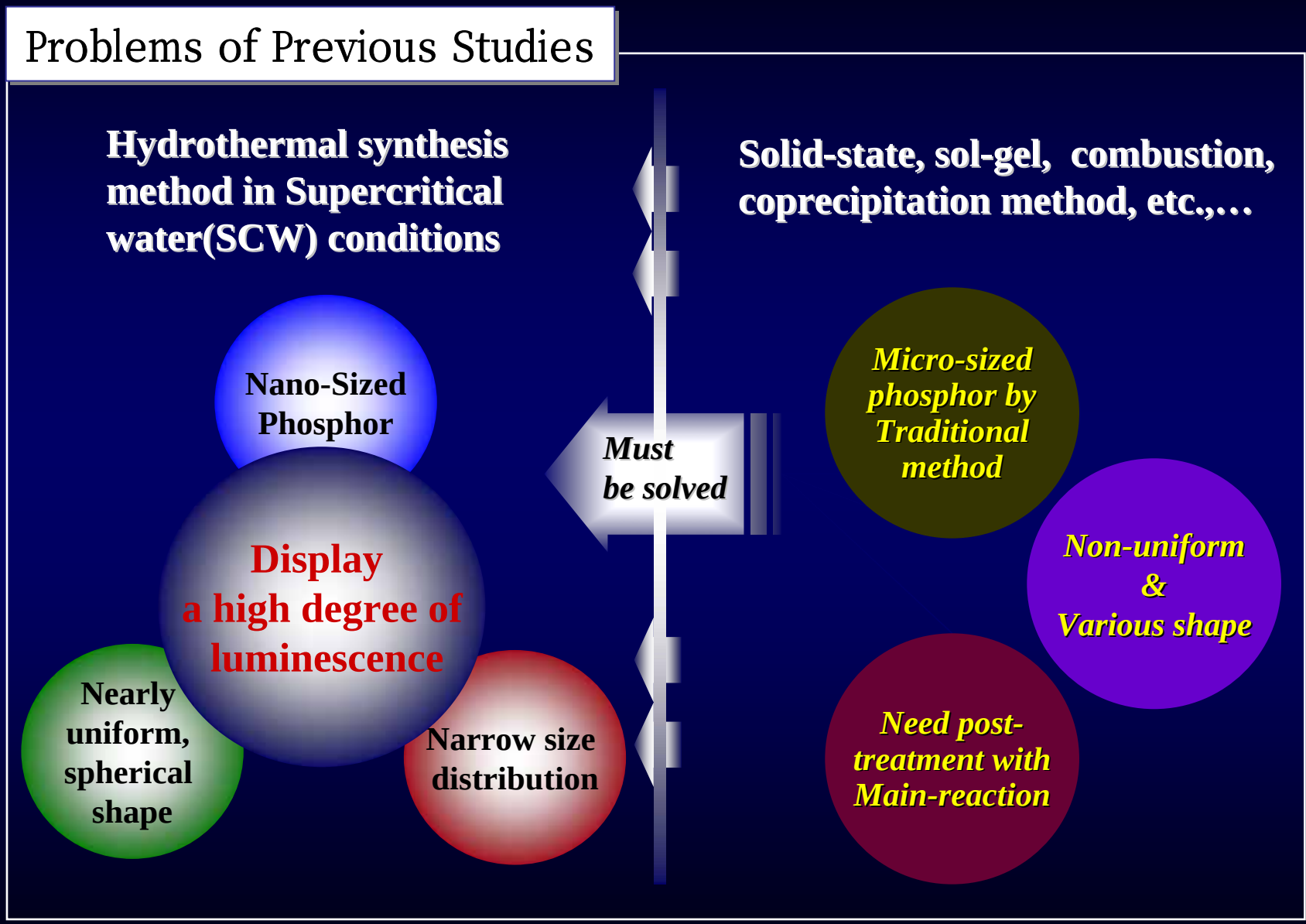
Application field



Problems of Previous Studies

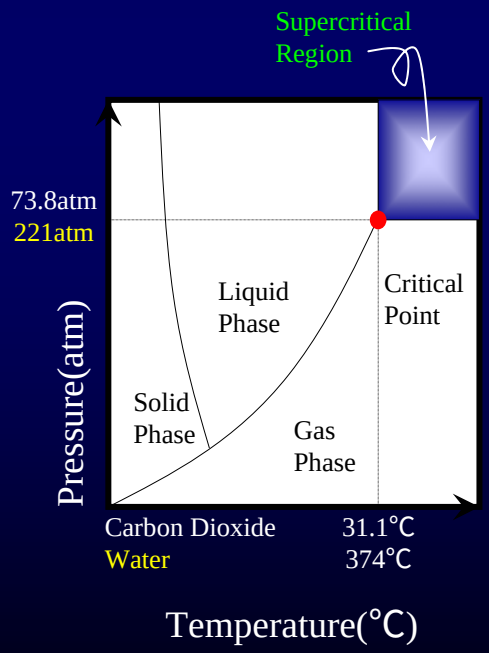


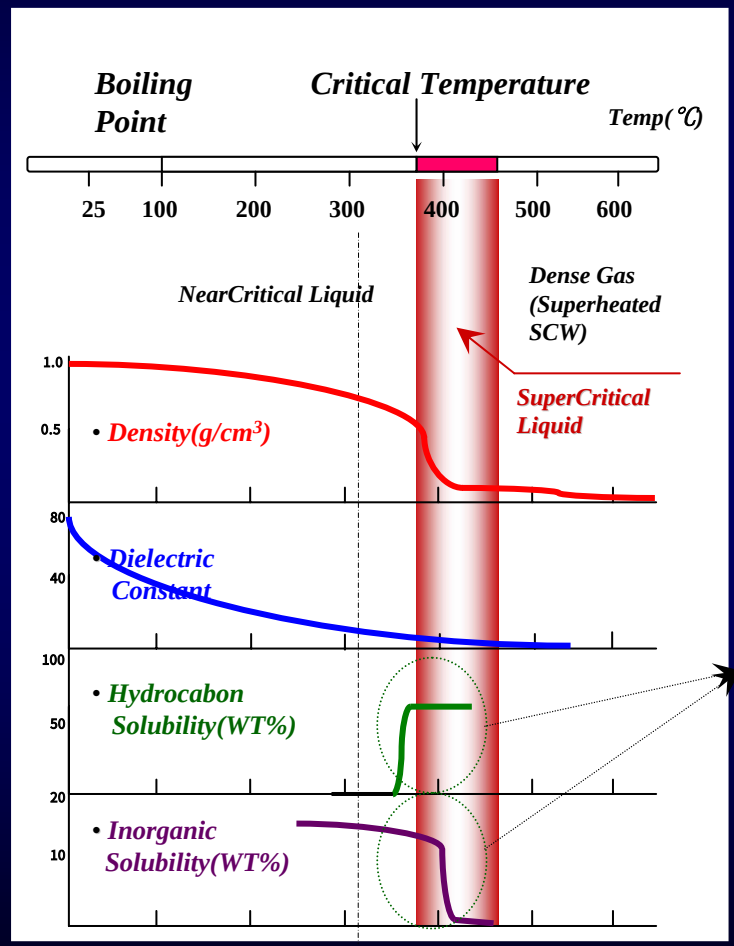
Property of Supercritical water(SCW)

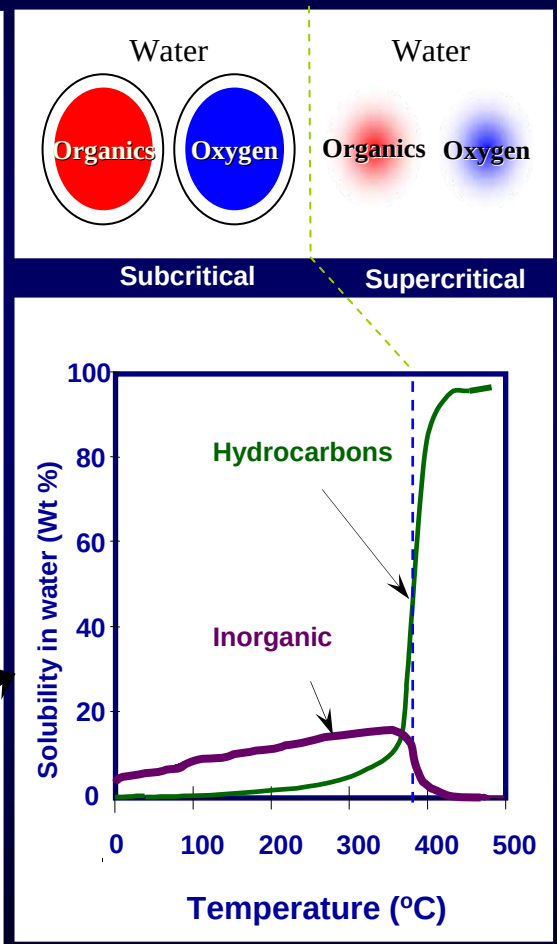
- **Supercritical fluid**

Properties

- **liquid like**
  density & solvent power
- **gas like**
  viscosity & diffusivity



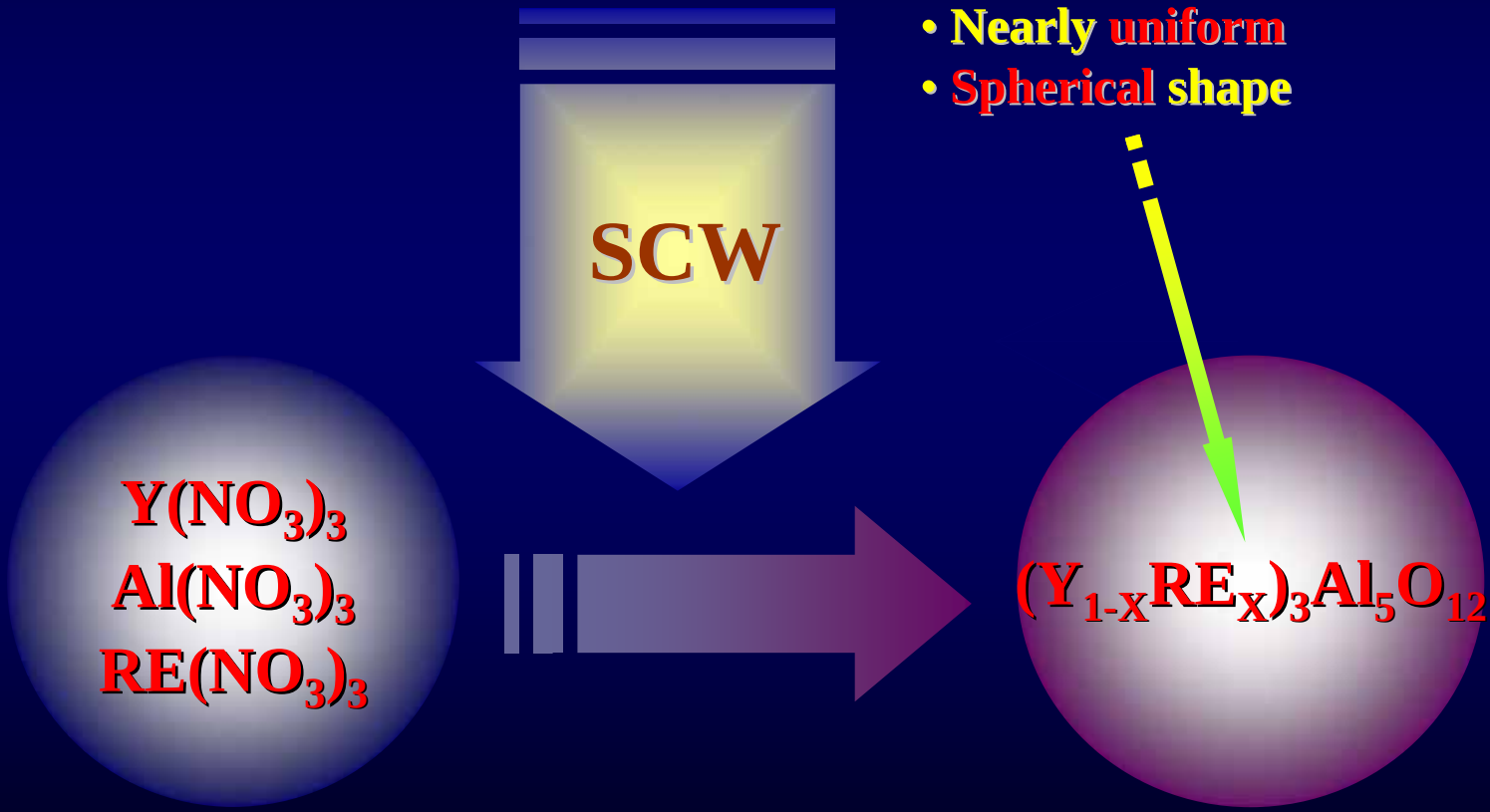




Supercritical Water

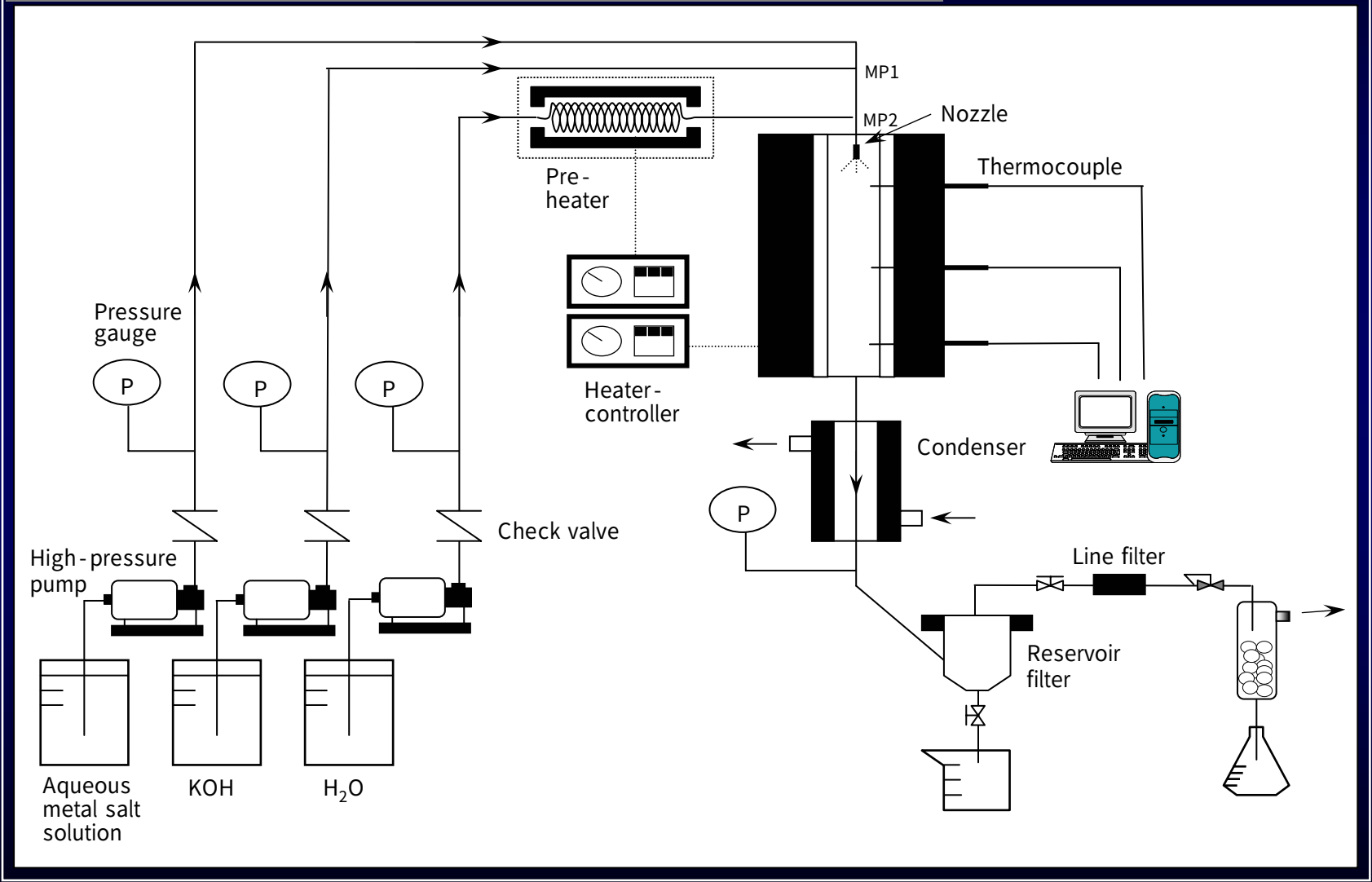
Chemical reactions

- No need of further treatment
- Crystalline state
- Nano-sized particles
- Nearly uniform
- Spherical shape

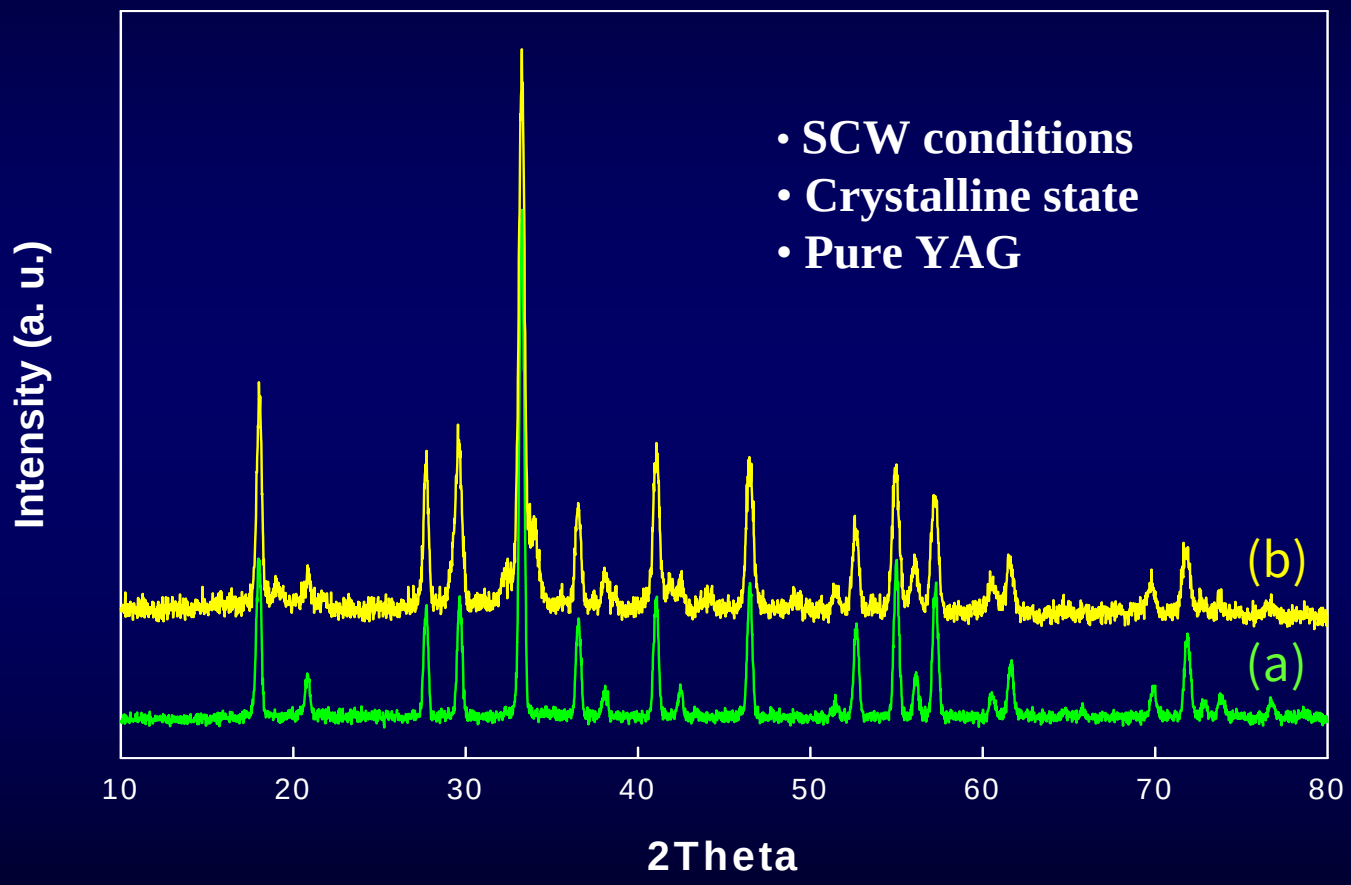


RE(Rare earth; Tb, Eu)

Experimental apparatus (Continuous system)



YAG:Tb (10 at. %) phosphor

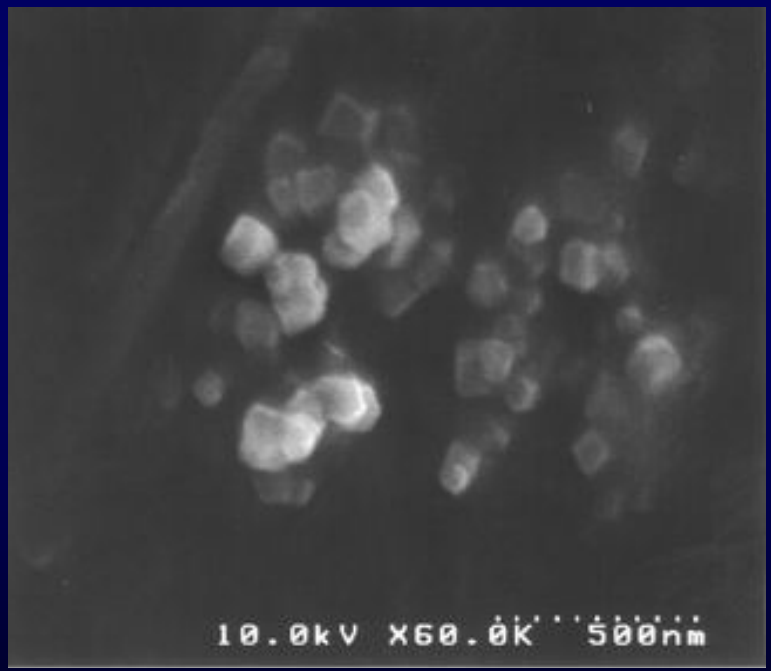


XRD patterns of YAG standard peak(a) and YAG:Tb(b)

YAG:Tb (10 at. %) phosphor

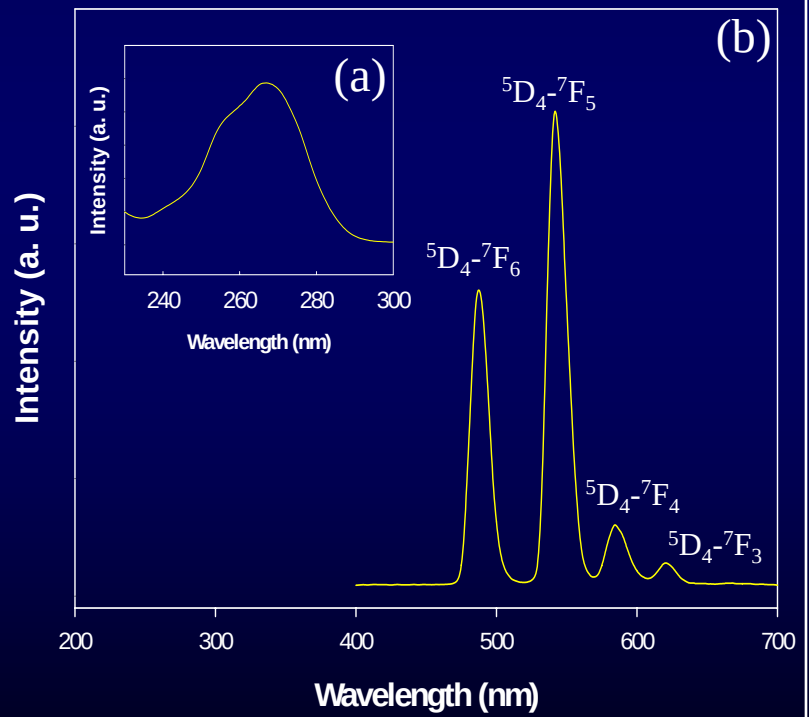
- **Synthesis of nano-sized YAG:Tb phosphor in SCW conditions**

Cubic-like YAG:Tb nanoparticles



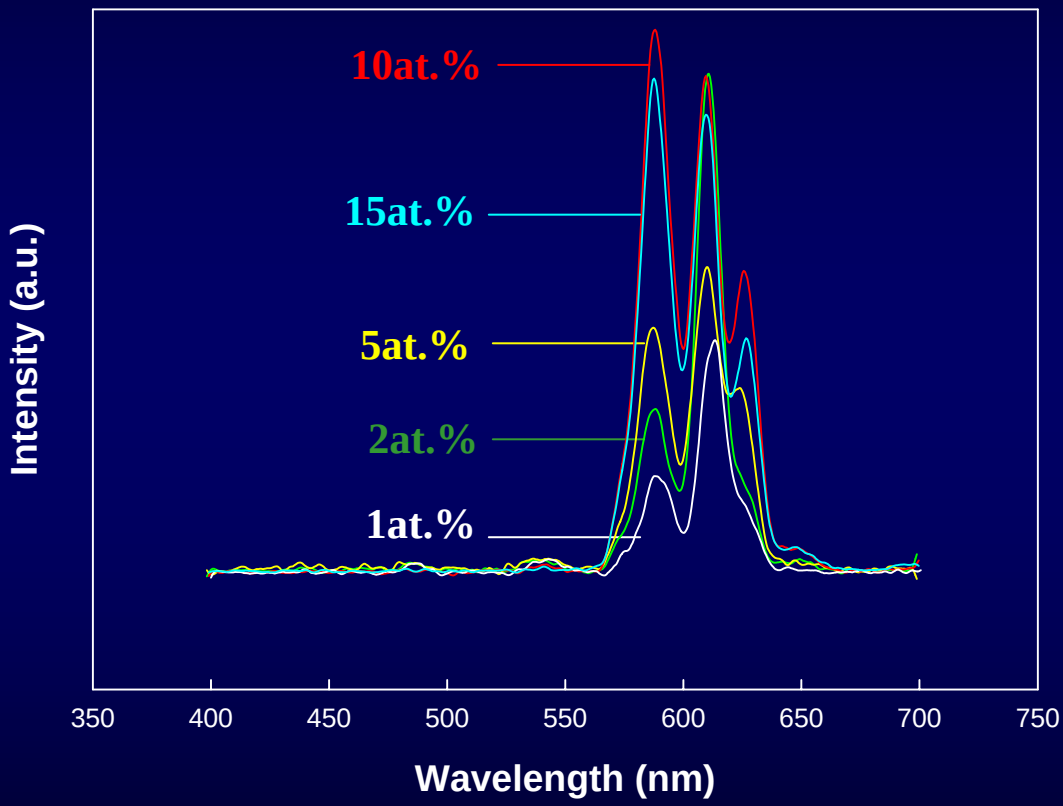
SEM image

Green spectral area



(a) Excitation(λ_m=540nm) &
(b) Emission(λ_m=267nm) spectra

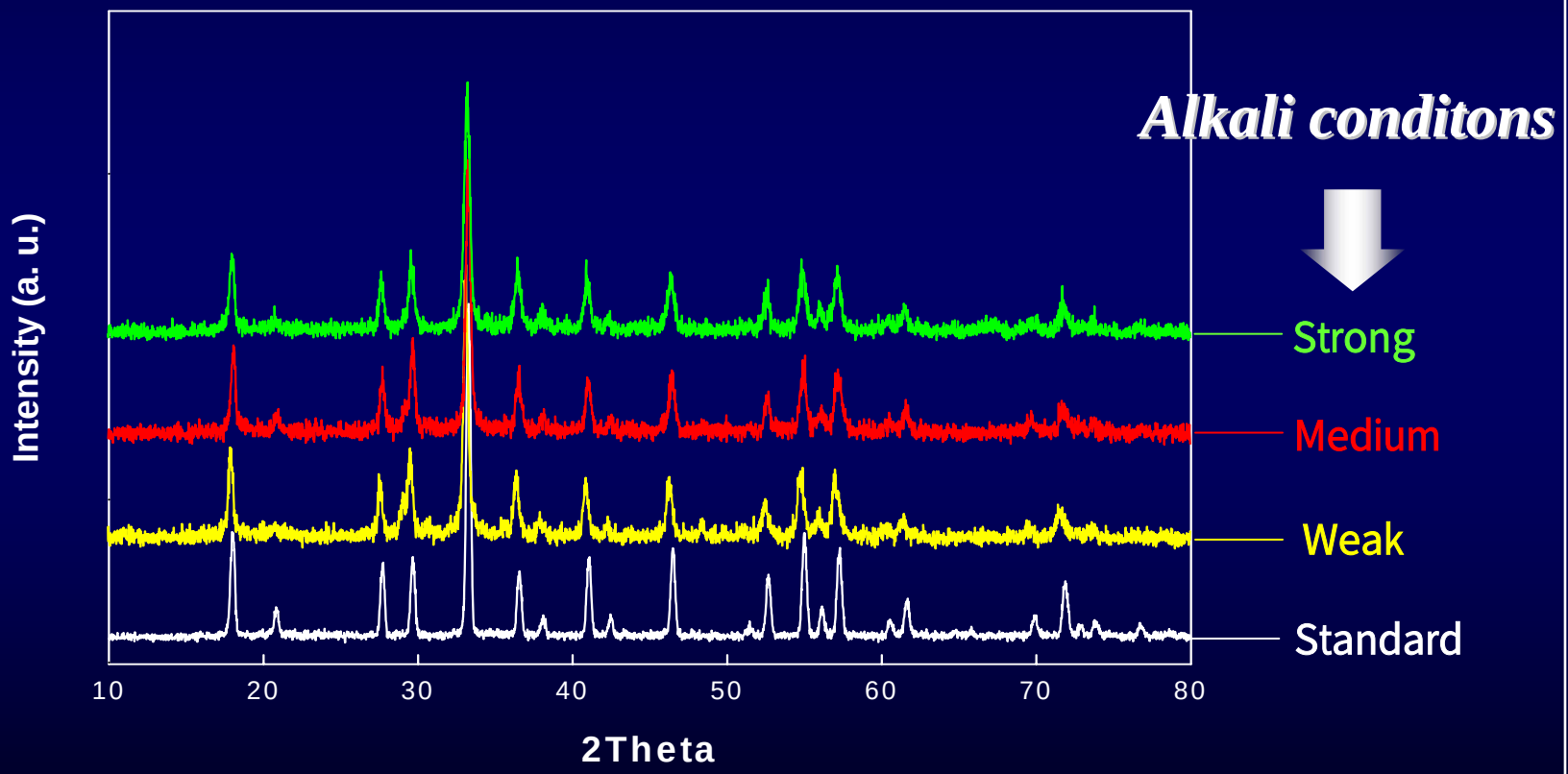
YAG:Eu phosphor with different Eu concentration



The highest intensity at Eu 10 at.% concentration

Characteristics of YAG:Eu phosphor with different pH

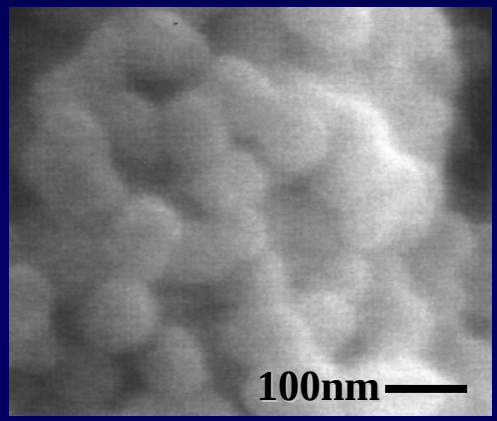
XRD of YAG:Eu(10 at.%) phosphor with different pH



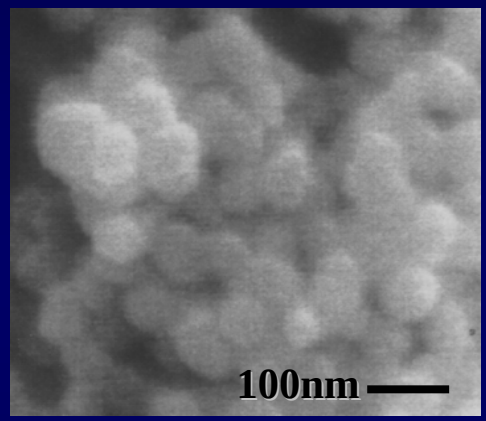
XRD* patterns with different *pH

Characteristics of YAG:Eu phosphor with different pH

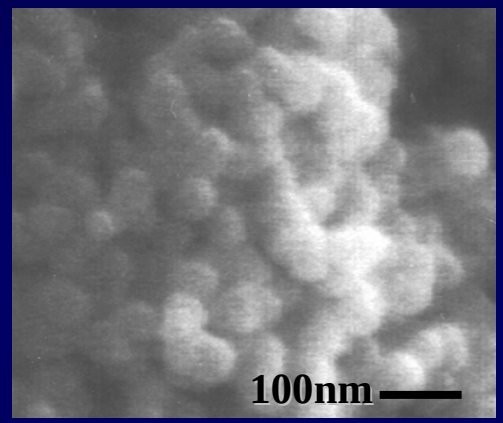
SEM of YAG:Eu(10 at.%) phosphor with different pH



(a) pH=weak



(b) pH=medium

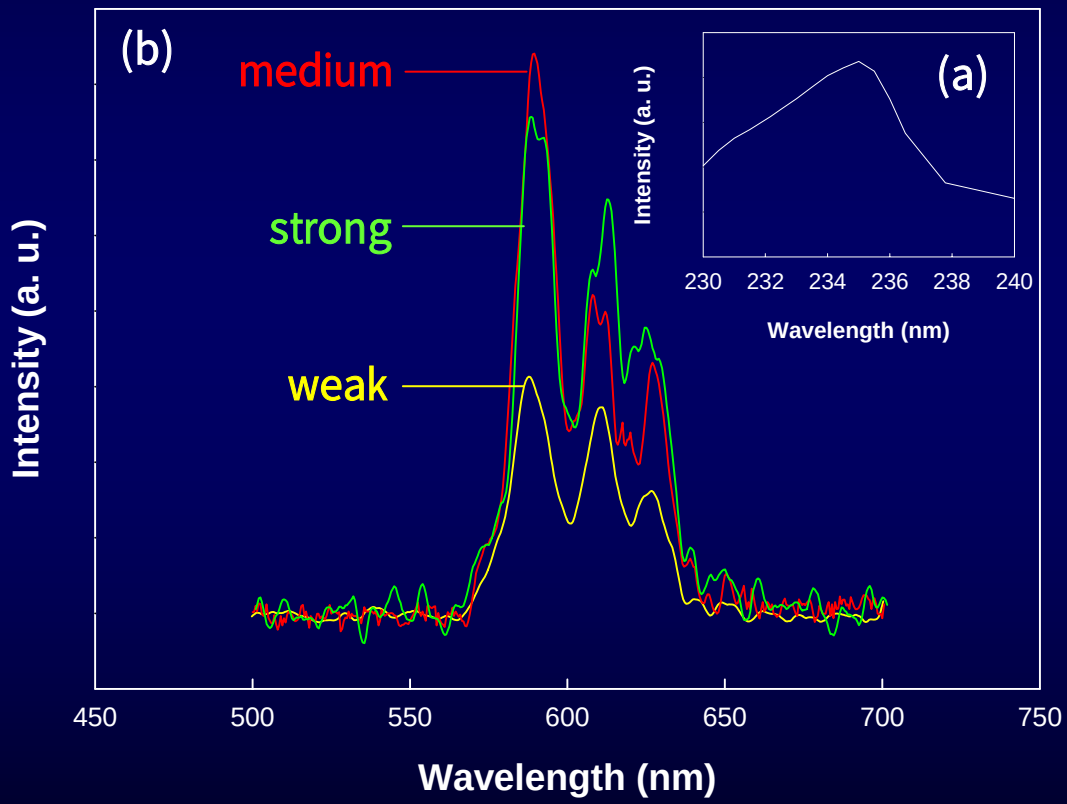


(c) pH=strong

- With a rise in the pH, the particles size decreased (Increase of solubility)
- Their morphology became nearly spherical
- The particles size was about 50-100nm

Characteristics of YAG:Eu phosphor with different pH

PL of YAG:Eu(10 at.%) phosphor with different pH

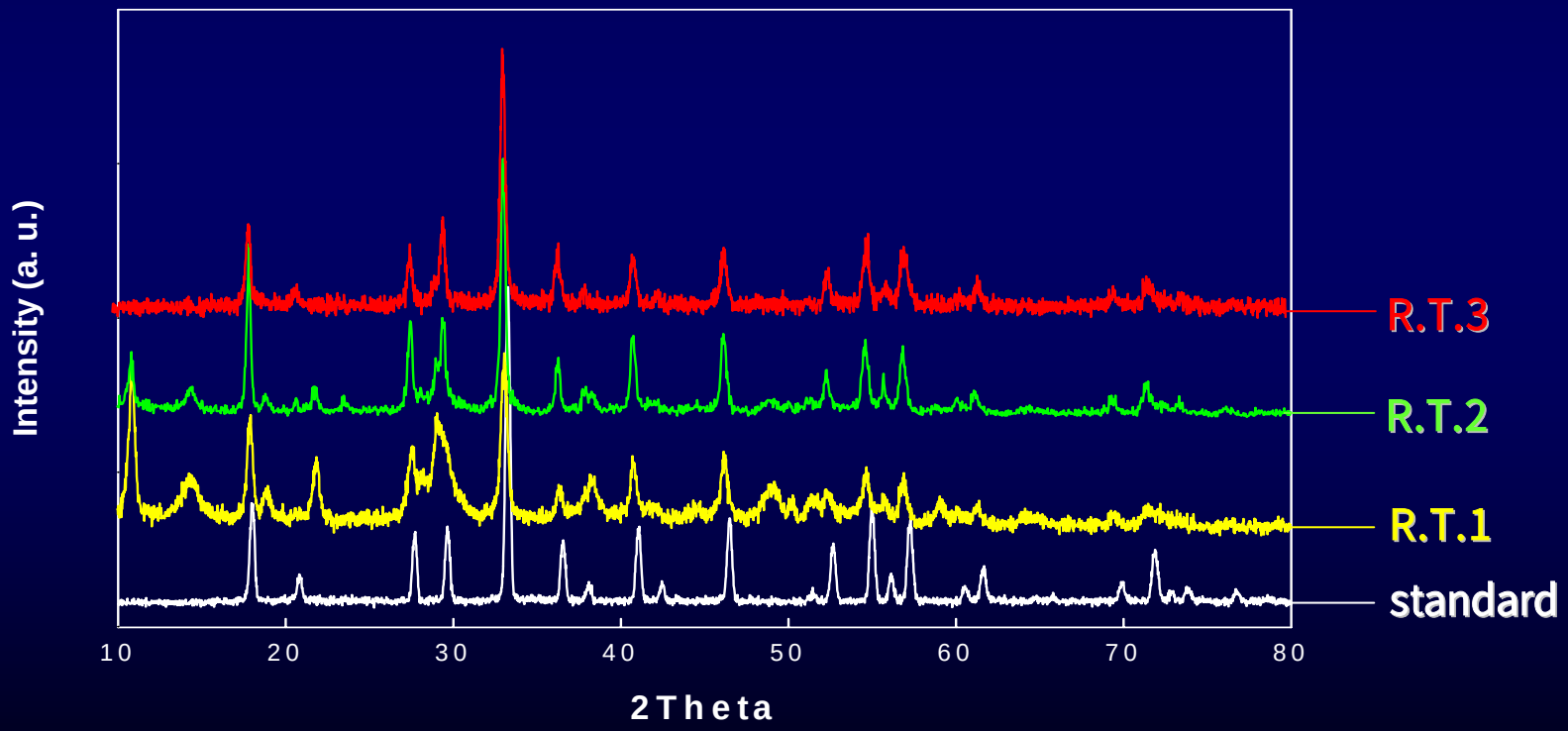


(a) Excitation(λ_m=590nm) & Emission(λ_m=234nm) spectra

YAG:Eu phosphor with different Reaction Time

XRD of YAG:Eu(10 at.%) phosphor with different R.T.

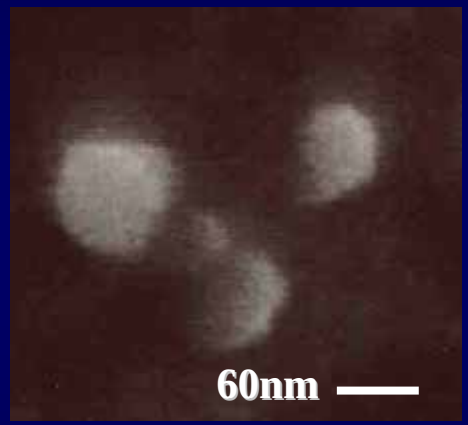
- *R.T.1 & R.T.2 : By-product(YAM, YAP), YAG*
- *R.T.3 : Pure YAG*



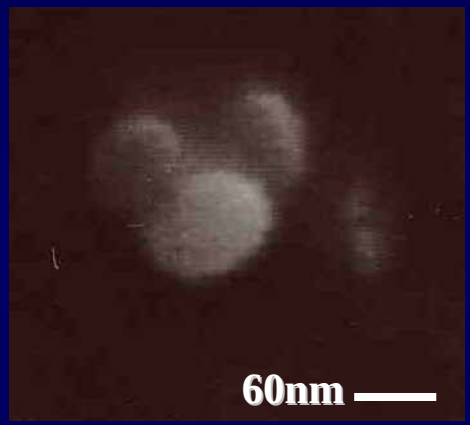
XRD* patterns with different *R.T

YAG:Eu phosphor with different Reaction time

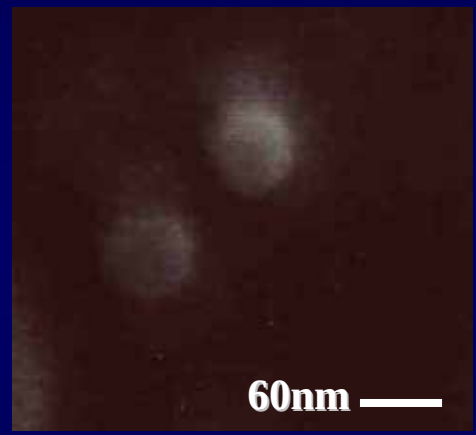
SEM of YAG:Eu(10 at.%) phosphor with different R.T.



(a) R.T.1



(b) R.T.2

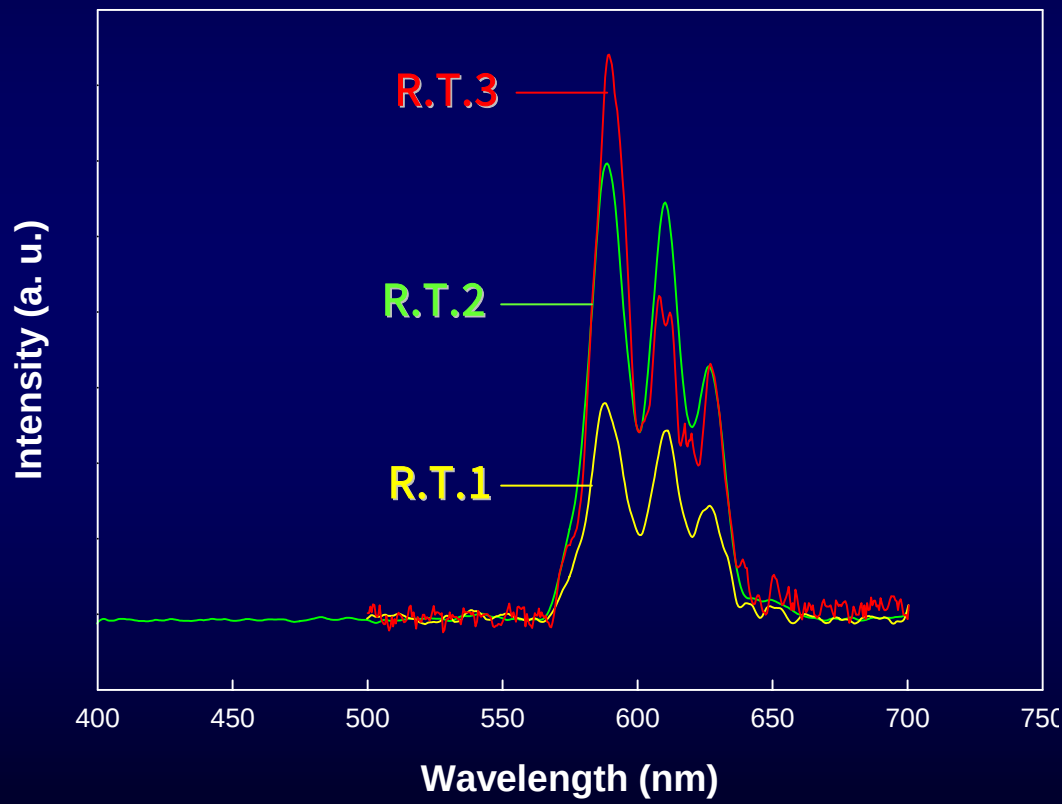


(c) R.T.3

- With a rise in the R.T., the particles size decreased
- The morphology became nearly spherical with an increase in R.T.
- The particle size was about 50-100nm

YAG:Eu phosphor with different Reaction time

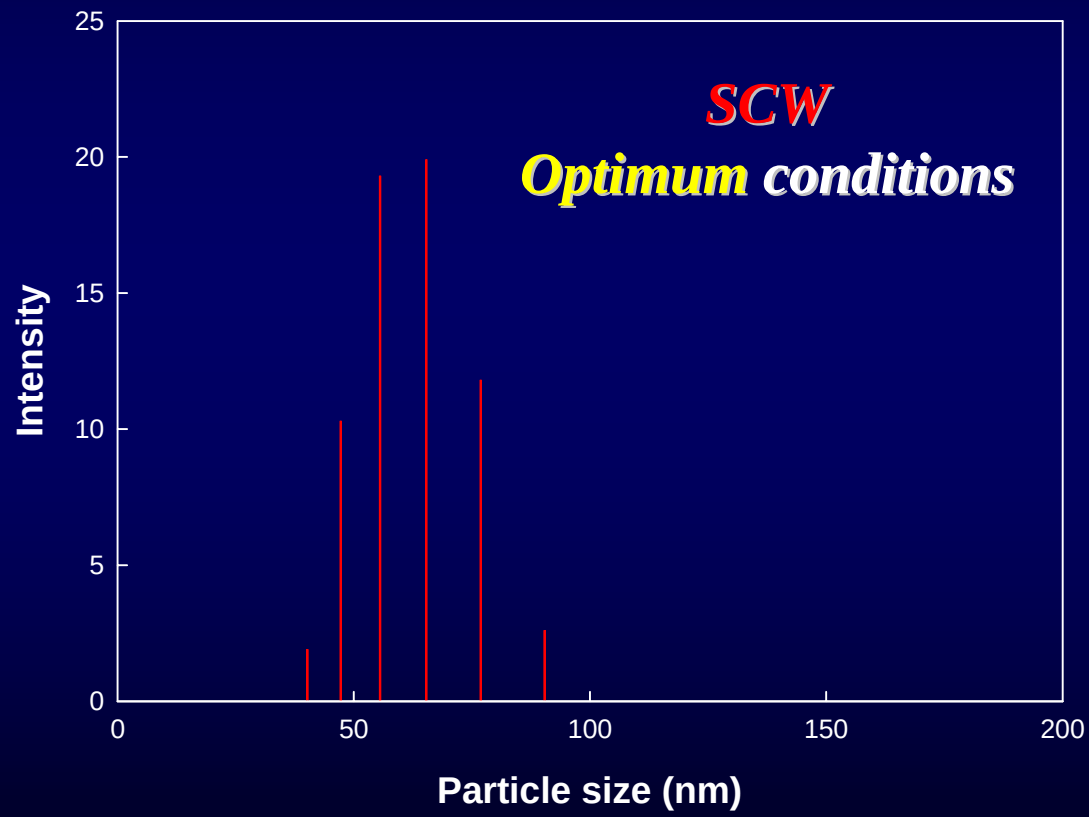
PL of YAG:Eu(10 at.%) phosphor with different R.T.



Emission(λ_m=234nm) spectra

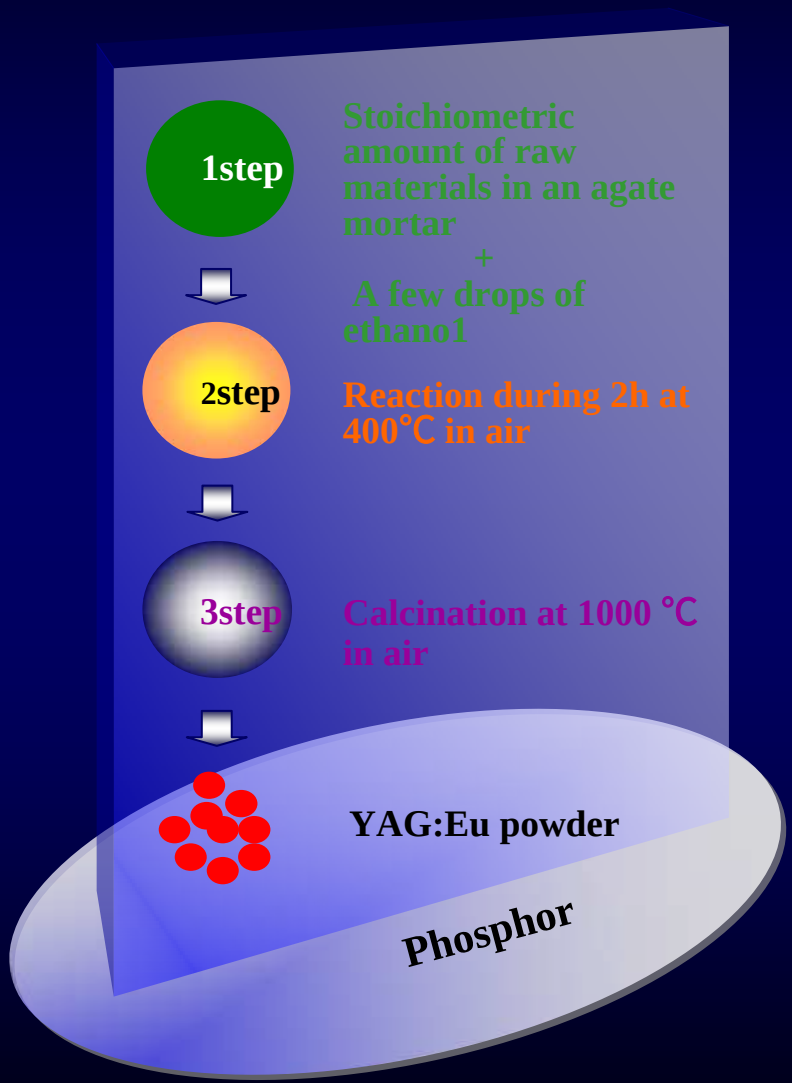
YAG:Eu phosphor

Narrow size distribution in case of SCW method



Size distribution of prepared particles

Synthesis of YAG:Eu phosphor by solid-state method



YAG phase reaction

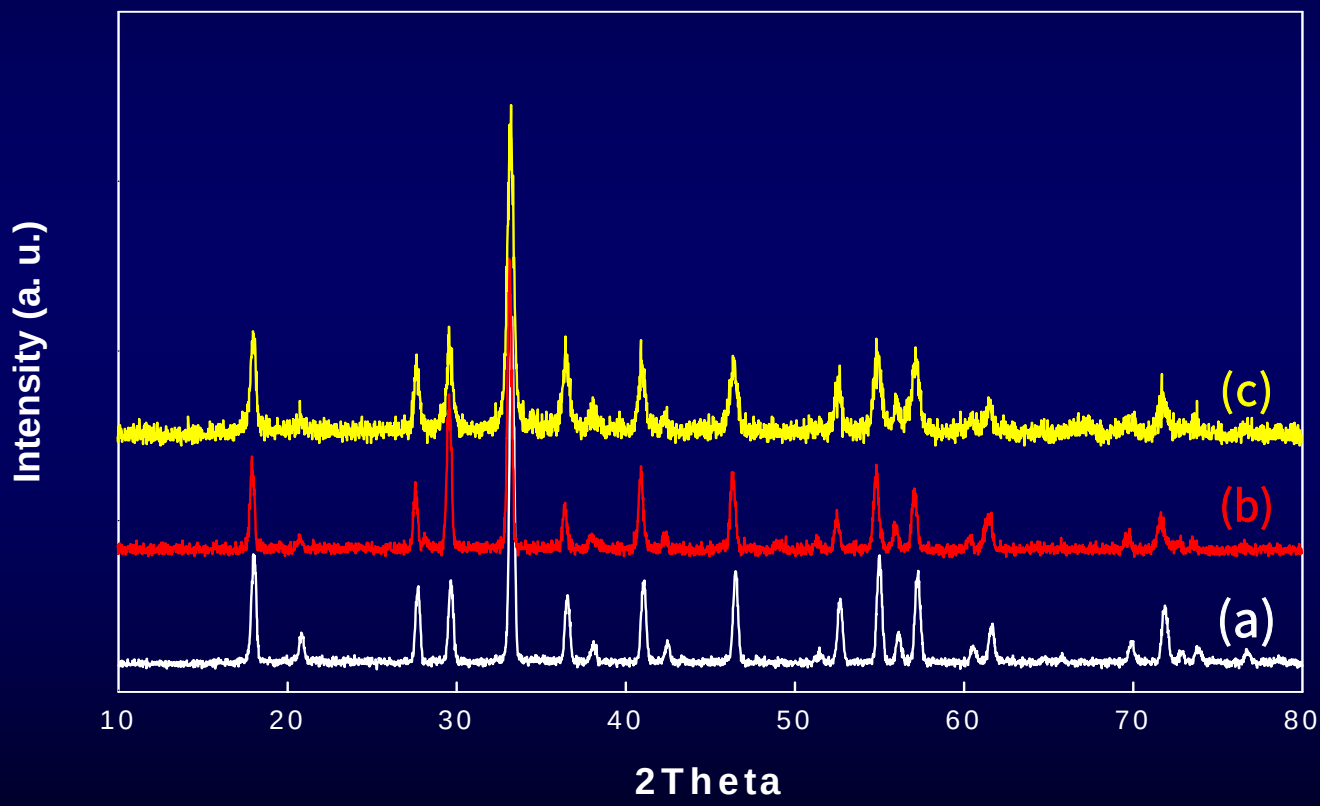
1. $2Y_2O_3+Al_2O_3 \rightarrow Y_4Al_2O_9$
(YAM; Yttrium aluminum monoclinic)

2. $Y_4Al_2O_9+Al_2O_3 \rightarrow 4\ YAlO_3$
(YAP; Yttrium aluminum perovskite)

3. $3YAlO_3+Al_2O_3 \rightarrow Y_3Al_5O_{12}$
(YAG; Yttrium aluminum garnet)

YAG:Eu phosphor

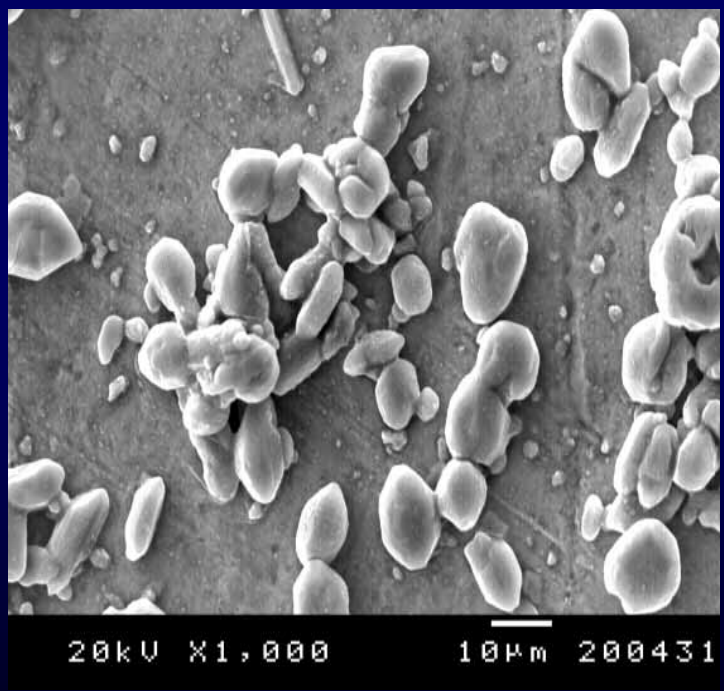
Synthesis of $Y_{2.99}Al_5O_{12}:Eu_{0.01}$ phosphor by solid-state method

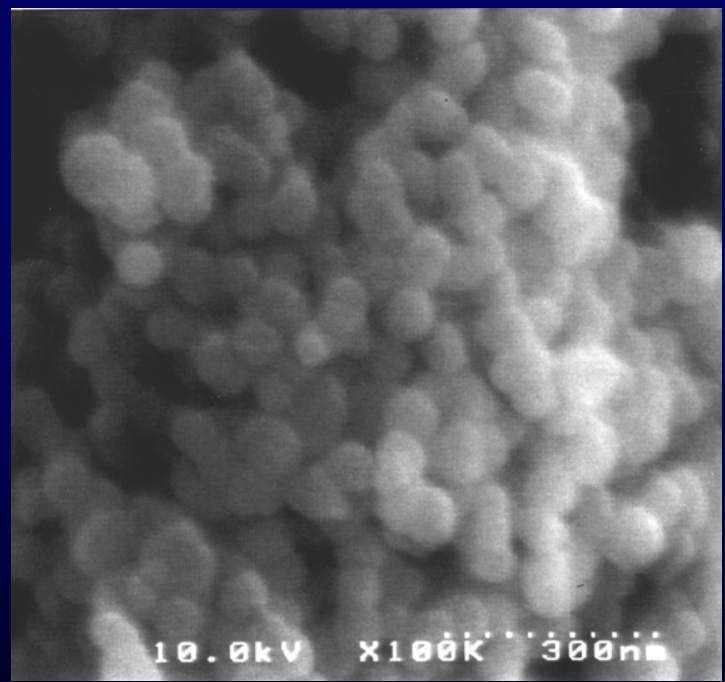


XRD patterns of YAG standard peak(a) and YAG:Eu(b; batch, c; continous)

SEM image of YAG:Eu phosphor

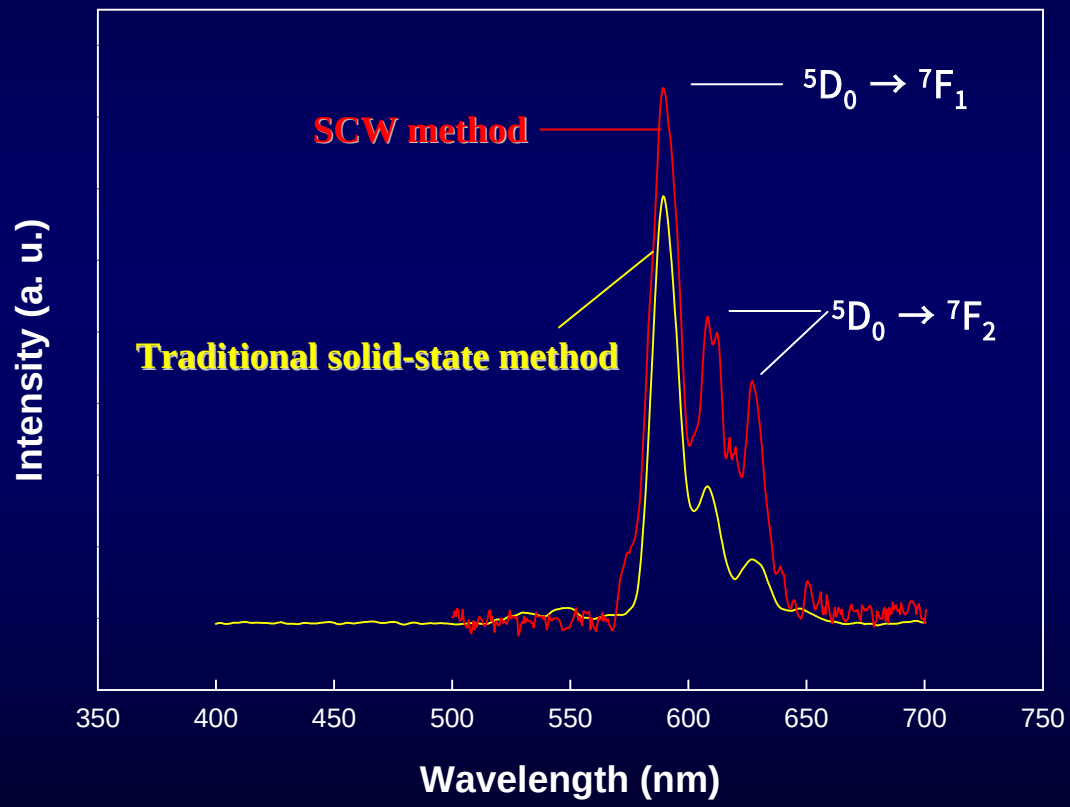
- The particles size of YAG:Eu powders synthesized in SCW(50nm) conditions was much smaller than that of the powders from the Solid-state method(1-10μm)
- Phosphor by SCW method became nearly spherical. On the other hand, the particles of solid-state method was irregular micro-sized particles





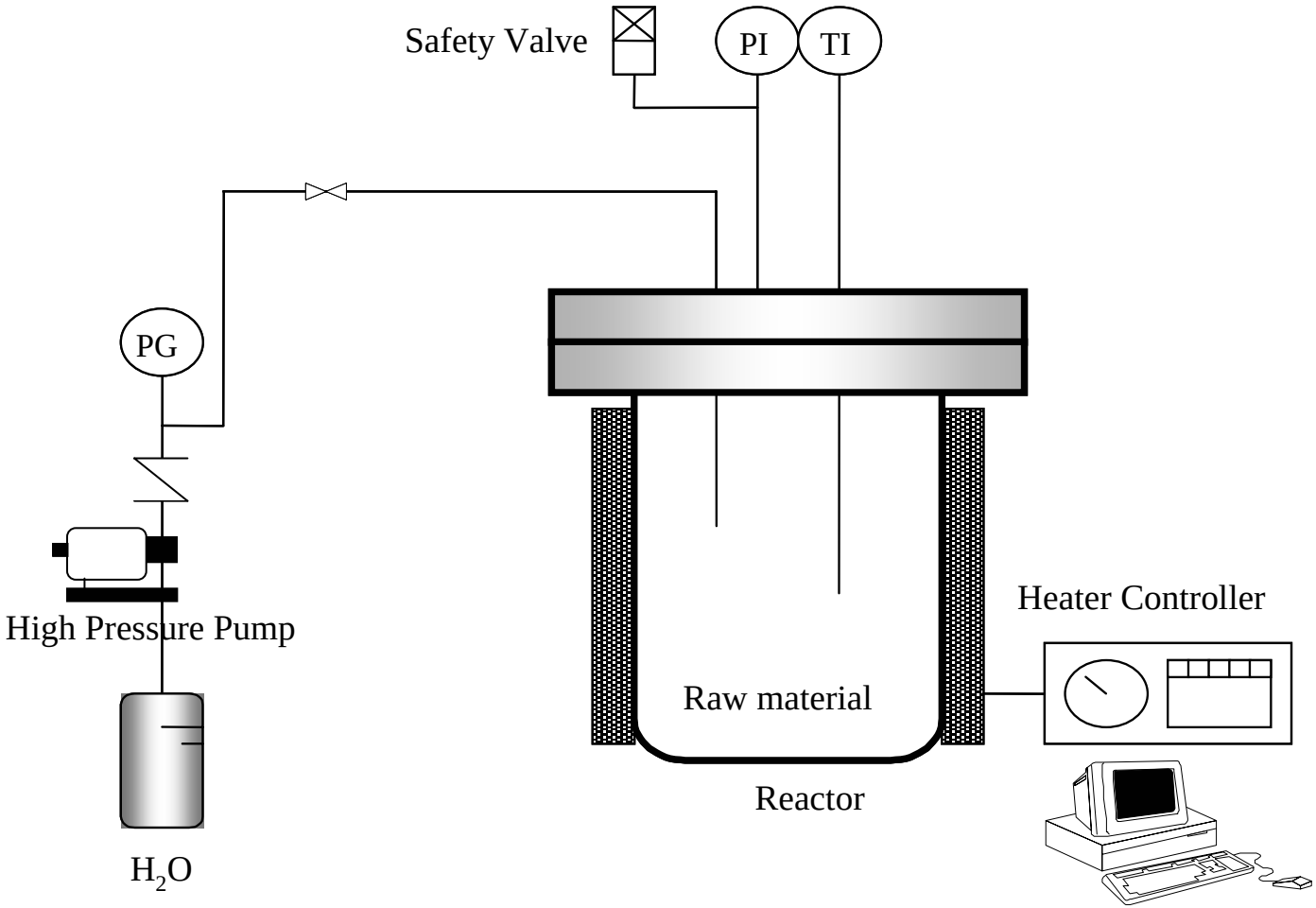
YAG:Eu phosphor

- **PL intensity was stronger than solid-state method**



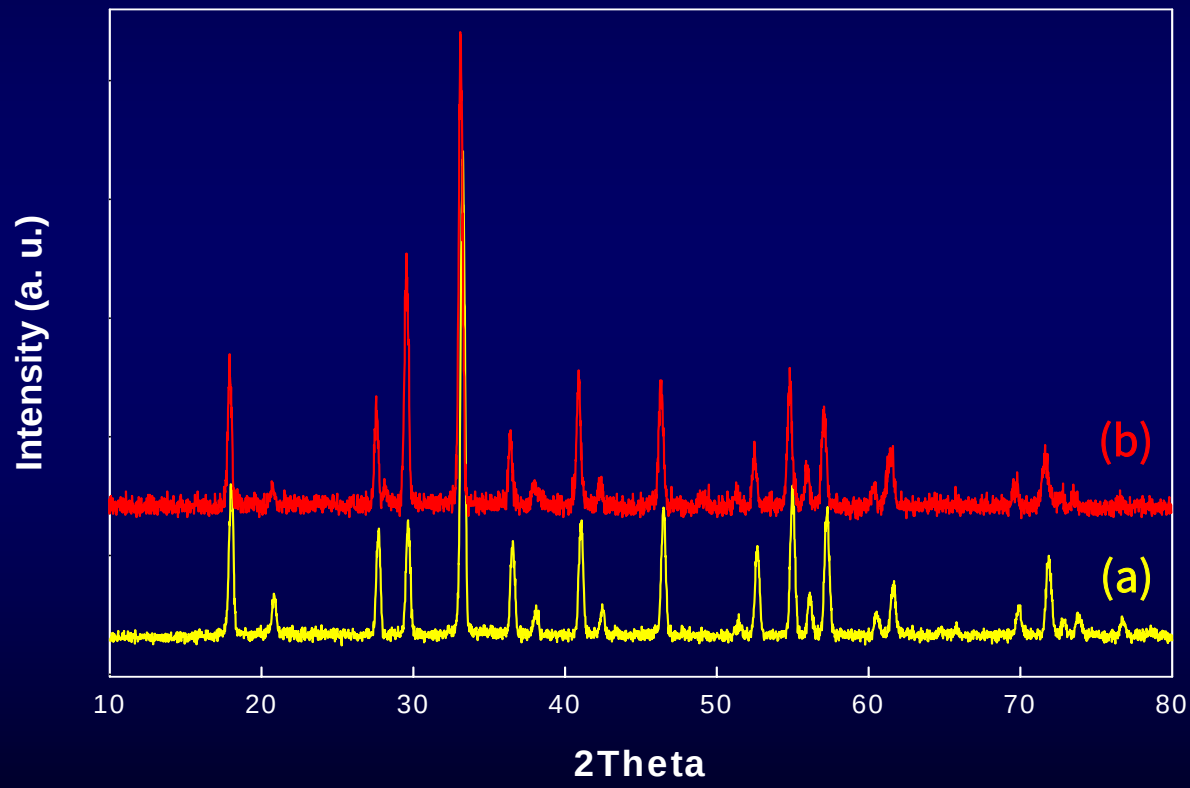
Comparison of Emission (λ_m=234nm) spectra

Experimental apparatus (Batch system)



YAG:Eu phosphor

Synthesis of YAG:Eu(10 at.%) phosphor in SCW (Batch reactor)



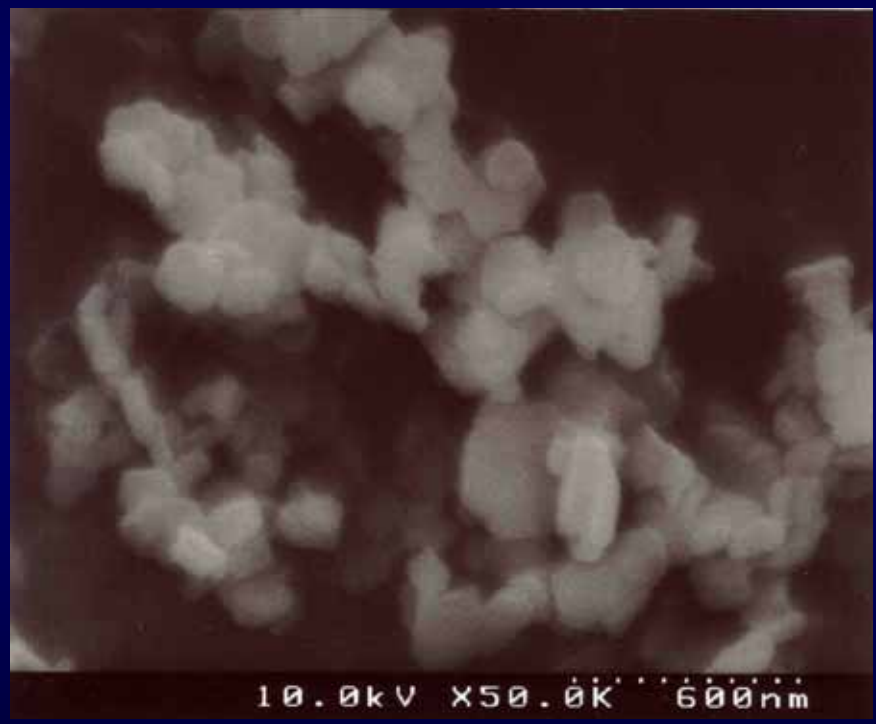
SCW conditions at batch system

(R.T.=4h)

XRD patterns of YAG standard peak(a) and YAG:Eu(b)

YAG:Eu phosphor with different Reaction time

SEM of YAG:Eu(10 at.%) phosphor at batch system



- **The particle size was about 50-100nm**
- **Several shape of particles were mixed**

Conclusions

Synthesis of Nano-sized Phosphors in Supercritical Water(SCW) conditions

- We have discovered that SCW can be used to produce nano-sized phosphor particles in continuous reactor without need further treatment (calcination).
- Unlike the traditional method, the SCW method can reduce production time for phosphor because the product does not need post-treatment and long time reactions
- The syntheized nano-sized particles (50-100nm) were pure YAG with crystal structure.
- Also, morpology of phosphors could be controlled by the SCW condition for nearly uniform, spherical shape and narrow size distribution.
- Therefore, the synthesized phosphors have strong luminescent properties in no need of further treatment.

Acknowledgements

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