Institute of Technical Sciences of SASA

SYNTHESIS OF THE NANOSTRUCTURED YAP:Ce *VIA* SPRAY PYROLYSIS BY POLYMERIC PRECURSOR SOLUTION

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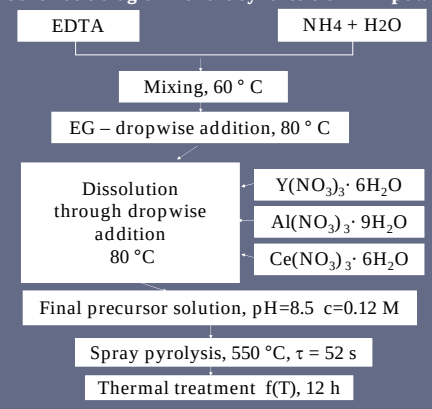
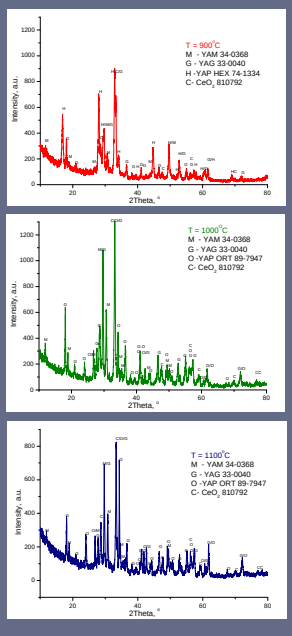
Abstract The yttrium aluminum system (Y_2O_3-Al_2O_3) includes three compounds: yttrium aluminum garnet ($Y_3Al_5O_{12}$, YAG), yttrium aluminum perovskite ($YAlO_3$, YAP) and yttrium aluminum monoclinic ($Y_4Al_2O_9$, YAM). Doped with Ce YAP and YAG phases are well known optical materials used as a fast scintillators for synchrotron X-ray experiments. Synthesizing single YAP phase is difficult even through wet chemical processing because of the possible allocations of other phases. Here, we tried to synthesize fine powders of $YAlO_3$:Ce^{3+} (5 at%) *via* spray pyrolysis of polymeric precursor obtained by dissolving the corresponding nitrates in ethylenediaminetetraacetic acid (EDTA) and ethylene glycol (EG) solution. Aerosol droplets are decomposed at 550 °C in argon atmosphere. In order to get a pure YAP:Ce phase as-prepared particles were additionally thermally treated in the range from 900 °C to 1100 °C for 12 hours in the air atmosphere.

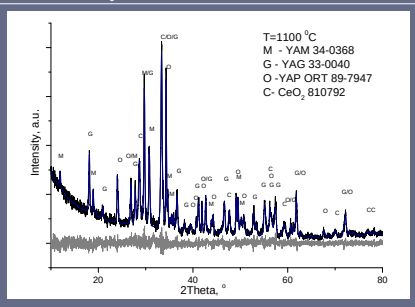
Schematic diagram for the synthesis of YAP powder





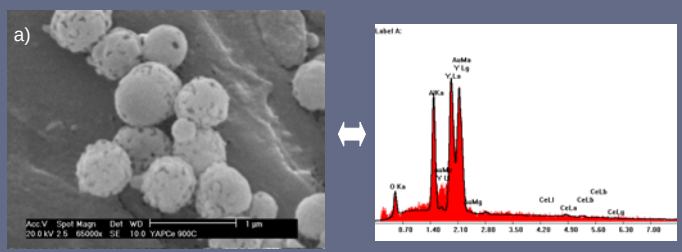
XRD analyses imply changeable phase compositions in thermally treated powders. The presence of a hexagonal YAP phase (*P63/mmc*) is observed at 900°C in ~70 wt%, while its orthorhombic modification is obtained at higher temperatures.
With the rise of temperature, the content of the YAG phase increases from ~ 10 to 50 wt%.
In all samplesYAM and cerianite phase are also present as minority ones.

Example of the structural refinement is presented for the powder thermally treated at 1100°C.

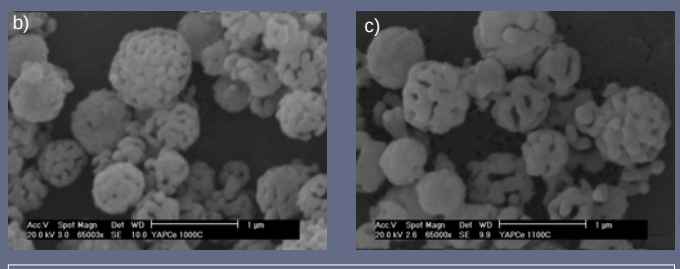


Microstructural parameters of determined phases (obtained through Rietveld refinement using Topas Academic software)

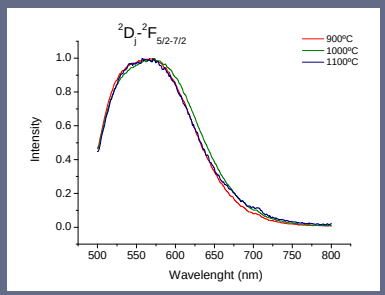
	900 °C, Gof = 1.59	1000 °C, Gof= 1.14	1100 °C, Gof = 1.12
YAP hexagonal [74-1334] S.G. P63/mmc	C.S. (nm) = 35(4) a(Å)=3.6(7) c(Å)=10.5(0)		
YAP orthorhombic [89-7947] S.G. Pbnm		C.S. (nm) = 269(0) a(Å) = 5.1(8) b(Å) = 5.3(2) c(Å) = 7.3(7)	C.S. (nm) =215(1) a(Å) =5.1(8) b(Å) =5.3(2) c(Å) = 7.3(7)
YAG cubic [33-004] S.G. Ia-3d	C.S. (nm) = 56(0) a(Å) = 12.0(3)	C.S. (nm) = 84(9) a(Å) = 12.0(4)	C.S. (nm) = 84(9) a(Å) =12.0(3)
YAM Monoclinic [34-0368] S.G. P21/a	C.S. (nm) = 19(7) a(Å) = 7.3(5) b(Å) = 10.5(9) c(Å) = 11.0(4)	C.S. (nm) = 76(4) a(Å) =7.3(8) b(Å) = 10.4(5) c(Å) =11.1(5)	C.S. (nm) = 72(6) a(Å) =7.3(7) b(Å) = 10.4(4) c(Å) =11.1(3)
Cerianite cubic [81-0792] S.G. Fm-3m	C.S. (nm) = 18(1) a(Å) = 5.49(9)	C.S. (nm) = 18(9) a(Å) = 5.4(0)	C.S. (nm) = 29(1) a(Å) = 5.4(0)



Highly spherical particle morphology is revealed with the SEM analyses. Particles are agglomeration-free and have high porosity. Volume precipitation is predominant. Temperature increase leads to the separation of the primary particles. Size of the secondary particles ranged from 200-800 nm; the mean particle size is around 350 nm. EDS analysis confirms required cations ratio (Y:Al ~1:1).



SEM/EDS analyses of YAP:Ce^{3+} powders obtained *via* spray pyrolysis – thermally treated at: 900 (a), 1000 (b) and 1100 °C (c)



Photoluminescence emission spectra indicates wide green-yellow emission band with the maximum at 570 nm. This feature can be tentatively ascribed to the ^2D-$^2F_{5/2-7/2}$ electron transition of Ce^{3+} ions in the YAG matrix.

Conclusion Highly spherical, submicronic in size and agglomerated-free particles were obtained *via* spray pyrolysis method using polymeric precursor solution. Independently of the additional thermal treatment applied multiphase composition is confirmed in all samples. Target hexagonal YAP phase (70 wt%) is observed after thermal treatment at 900°C (12h), while additional rise of the temperature stabilizes the YAG phase generation and Ce^{3+} accommodation in it. Further synthesis optimization steps will be performed towards stabilization of kinetically favored pure YAP phase formation.

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