STUDY OF THE XRPD MICROSTRUCTURAL PARAMETERS

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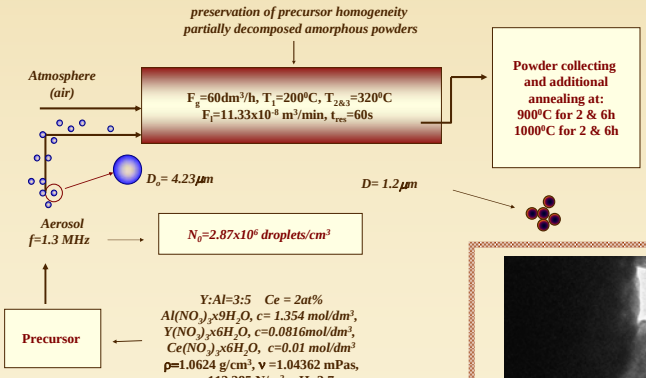
Abstract

Comparative analysis for the YAG:Ce^{3+} microstructural parameters is performed through Fullprof and Koalariet-XFit programs. Both programs are based on Rietveld method of structural refinement where fitting is done by convolution and corrections of XRD peak width for instrument broadening through either using the reference specimen (Fullprof) or the fundamental parameters approach (Koalariet-Xfit).

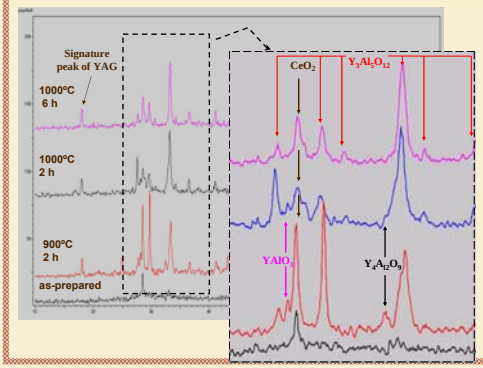
YAG:Ce^{3+} powder is obtained *via* low temperature aerosol route at 320°C followed with post annealing treatment at 900 and 1000°C. Besides targeting cubic garnet structure $Y_3Al_5O_{12}$ (YAG), the presence of either perovskite $YAlO_3$ (YAP) or monoclinic $Y_4Al_2O_9$ (YAM) phase is detected. Since garnet phase represents a very promising host phosphor material being doped with rare-earth ions, the most important criteria determining its applicability in various optical devices is the uniform distribution of the luminescent center in the host lattice as well as the cubic YAG phase crystallinity.

Comparison of data obtained for same the sample through usages of the above mentioned XRPD analyses gives insight of the validity of the refined parameters.

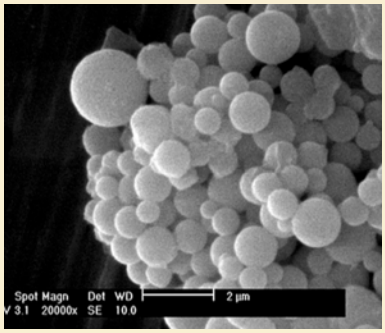
Low temperature aerosol decomposition



Phase development



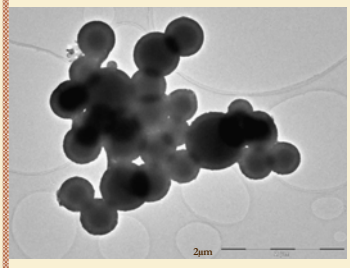
SEM (XL30ESEM Philips)

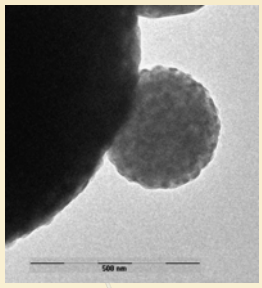


Spherically shaped and submicron sized as prepared particles

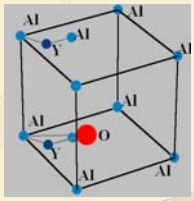
D_{mean}=0.7μm mm

TEM (Tecnai 20 Philips)





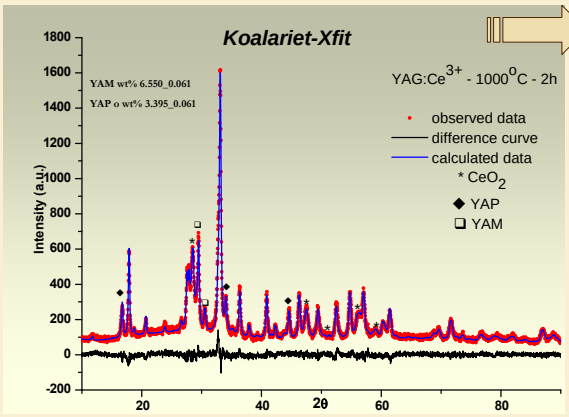
Composite inner particle structure



Al-O: 1.93189 1.76415
Y-O: 2.32885 Y-Al: 3.01420

Structure refinement

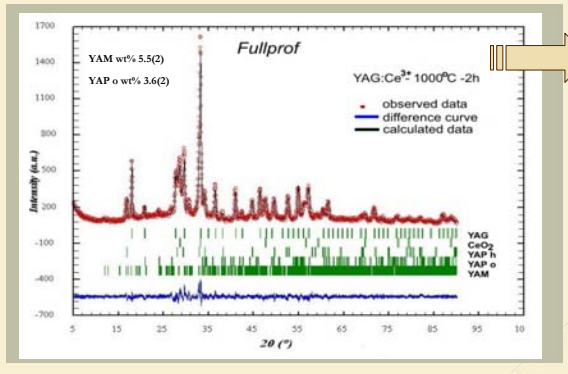
(XRPD data: step 0.02, time 15s)



Koalariet program (fundamental parameter approach): gof=0.914 rwp=7.433

background: polynomial function of the 8th degree

YAG Ia-3d cubic crystalline	YAG Ia-3d cubic inter-crystalline	CeO_2 Fm-3m cubic crystalline	CeO_2 Fm-3m cubic inter-crystalline	YAP P6 3/mmc hexagonal	
lp (Å): 12.0568_0.0001	12.0801_0.0001	lp: 5.4059_0.001	5.3933_0.0036	$lp_{1,2}$: 3.6847_0.001	lp_3:10.5003_0.0015
wt%: 47.988_0.461	12.110_0.587	12.110_0.587	1.011_0.116	15.723_0.173	
CS (Å): 647.25_38.53		126.56_15.52		460.47_22.39	
strain: 0.485_0.024	1.309_0.181	0.0001_0.1793	1.07008_0.17978	0.499_0.039	



Fullprof program: X^2=0.803 Rwp=15.3 R=5.86 Rexp=17.61

background: linear interpolation between 34 points

YAG Ia-3d cubic crystalline	CeO_2 Fm-3m cubic crystalline	YAP P6 3/mmc hexagonal	
lp (Å): 12.0454(7)	lp: 5.3955(3)	$lp_{1,2}$: 3.6742(3)	lp3: 10.4883(9)
wt%: 57.8(7)	12.3(2)	20.6(3)	
CS (Å): 373.58(0.12)	95.48 (0.04)	236.43(0.25)	
strain: 26.5226(0.0216)	25.3791(0.0052)	11.2298(0.0096)	

Conclusion

Fundamental parameter approach in Koalariet uses physical based models to generate the line profile shape so it enables recognizing of crystalline and intercristallyne domains presence in YAG and CeO_2 and gives more accurate insight in nanocrystaline particle nature.

Standard approach in Fullprof uses analytical-function fit (Tompson-Cox-Hastings pseudo-Voigt function) resulting in more correlated crystalline size and strain parameters determination where intercrystalline domains could not be considered.

Both programs allow to obtain structural and compositional information for all crystalline phases present in analysed sample but in the case of low weight percentages they could not be modelled.

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