

Nd, Cr, Ce: YAG NANO POWDER FOR SOLAR PUMPED LASER AND CRYSTAL SPECIFICATIONS

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ABSTRACT

Nd,Cr,Ce:YAG nano powders were synthesized via sol gel co precipitation method and commercial mixing method. Both powders were calcined at 900°C - 1100 °C. Further the crystals were subjected to vacuum sintering from 1700 °C - 1750°C for 8h - 20h. The effects of calcinations and sintering on the nano -structure was studied by SEM, EDAX, TEM, TGA-DSC. UV spectroscopy was studied to illustrate the optical properties of crystals. SEM morphology showed soft powder with spherical shape of particle size >90 nm as seen in the TEM images too. After sintering the crystal showed high compact with mainly one phase and very small in number and size of pores. The EDX analysis showed the %Y/Al= 0.6 near the theoretical value and the XRD sharp peak confirmed YAG formation. The TGA-DSC curve showed thermal behaviour of the crystal and temperature of formation for YAG was nearly 750 °C. The optical property was studied by UV spectroscopy. The absorbance maxima at 0.5 appeared around 300 nm and a band area of multiple peaks for Nd,Cr,Ce:YAG was illustrated.

Keywords: sol gel nano sphere powder, doped YAG, solar pumped laser, energy applications.

INTRODUCTION

Last decades rare-earth doped garnet materials used in medicine, optical, laser, and biotechnological processes [1, 2]. Different chemical methods are used for synthesis, such as sol gel, solid state, self-combustion, microwave, autoclave...etc. Sol gel process has considerable advantage compared to other synthesis methods because of better homogeneity, lower temperatures and controlled properties of final product [3, 4]. Ceramic yttrium aluminium garnet (Y_3 Al₅ O₁₂) has proved to be one of the most promising laser materials for many kinds of laser devices especially high power laser [5, 6]. YAG ceramic powder can be characterized by particle shape, size distribution, chemical composition, degree of agglomeration and purity [7].

Traditional solid-state reaction from oxide mixture requires repeated mechanical mixing and extensive heat treatment at high temperatures to obtain pure YAG, leading to uncontrolled nano structural features [8, 9]. The sol-gel processing co-precipitation method is one of the effective homogenous precipitations of colloidal spheres [10, 11].

Homogenous precipitation technique is a process of cation precipitation through thermal decomposition of urea [12, 13], in which (NH₂ CO NH₂) provides slow uniform production of anionic species, e.g. OH⁻ and CO₃²⁻, into the system through the hydrolysis of thermal decomposition products.

The fabrication of laser quality Nd:YAG, Ce:YAG and Nd,Cr,Ce:YAG by solid state reaction or sol gel coprecipitation with and without using sintering additives [TEOS] to obtain partial phase transformation mixture into garnet phase formation leads to homogenous densification [14]. In the energy field, ceramics are very important in the nuclear industry as fuel ceramics or for waste immobilization but also for electricity generation, transport and storage [15]. Conversely, ceramics are opaque and light hardly propagate through them. Development of new processing techniques for application of ceramics to the area of optical and photonic devices are recently taking relevance in optical technology. As well as some ceramics can be fabricated with small size particles, with homogenous grain size distribution and very low porosity may present transmittance values close to these of glass [16].

In the present work briefly describe the ceramic fabrication by sol gel method and commercial mixture reactive sintering wet chemical co-precipitation. Nd:YAG, Ce:YAG and or Nd,Cr,Ce:YAG nano powders. A study of the microstructure and inline absorbance optical properties for pumped laser applications.

1. MATERIALS AND METHODS

Materials

High purity materials 99.99% were bought such as Cr_2O_3 , Y_2O_3 , Nd_2O_3 , Ce_2O_3 , Acetic acid, Ethanol, Nitric acid, Tetra Ethyl Ortho silicate, (TEOS), Urea, [(NH₄)₂ SO₄] – Al(NO₃)₃.9H₂O- H₂O₂.

We used the sol gel method with autoclave technology and calcination at 1100°C 4-6h for preparation of five samples. Nd:YAG two crystals prepared by solid state and sol gel method, Ce:YAG prepared by commercial powder mixing, Nd,Cr,Ce:YAG prepared by sol gel, and autoclaved Nd,Cr,Ce:YAG at different time.

The morphology of prepared samples were studied by SEM, TEM,EDX, XRD, IR,TGA-DSC and activity of light absorption was measured by UV spectrophotometer.

Methods

Sol Gel method was used for preparations and autoclave technology aid, also the commercial mixing powder method was studied.

1st sample Nd:YAG...nano-powder prepared by sol gel method where Nd₂ O₃, Y₂O₃ were stoichiometric calculated and dissolved in nitric acid and Al(NO₃)₃.9H₂O used as Aluminium source. Urea and ammonium sulphate



used as starting materials. $[(NH_4)_2SO_4]/$ [mother salt] ratio was optimized. The mixture aged 2h and the solution was heated for one h at 80°C. The solution was quenched with ice water and the precipitated powder was washed by distilled water and ethanol, dried 2h at 80 °C and powder was the calcine at 1100 °C 4hr. Further sintering of powder at 1735°C – 1750 °C was subjected 20 h under vacuum.

 2^{nd} sample Ce-YAG (mixture)...Commercial Y₃Al₂O₁₂ and Ce₂O₃ were stoichiometric optimized and mixed together with acetone by grinding to homogenize the mixture. The sample was covered and calcined at 1100 °C 6h. The fine powder was then characterized. Further sintering of powder at 1735°C – 1750 °C was subjected 20 h under vacuum.

 3^{rd} sample Nd,Cr,Ce:YAG nano-powder prepared by sol gel method ...were cations stoichiometric calculated as $[Y^{3+}]:[Al^{3+}]:[Ce^{3+}]$ with molar ratio of (3-x):5:x. Al(NO₃)₃.9H₂O and Ce(NO₃)₃.6H₂O were added into solution with stirring. Cr₂O₃ was dissolved in nitric acid with addition of H₂O₂. The mixture was used as mother solution. Urea was added to solution according to molar ratio of [urea]/[total metal cations(Y³⁺+Al³⁺+Ce³⁺)]= 20. The (NH₄)₂SO₄ was added to the solution in mass ratio of [ammonium sulphate]/[mother salt].

The solution was then autoclaved 1h at 110°C. The precursor was washed with distilled water and ethanol then

dried at 80°C 12h. The powder was calcined at 1100°C 4h in air. The fine powder was then characterized. Further sintering of powder at 1735°C - 1750 °C was subjected 20 h under vacuum.

 4^{th} sample Nd,Cr,Ce:YAG was prepared by stoichiometric ratio of mixing Commercial $Y_3Al_2O_{12}$ with neodymium nitrate and cerium nitrate and chromium nitrate with stirring at 80 °C till homogeneity with addition of H_2O_2 and ozone gas. The mixture was then autoclaved 2h at100°C - 110°C. The precipitated powder was washed with distilled water and ethanol then calcined at 1100°C 6h in air. The fine powder was then characterized. Further sintering of powder at 1735°C – 1750 °C was subjected 20 h under vacuum.

2. RESULTS

New activities were needed to optimize the nanopowders synthesis process in order to obtain soft agglomerates that can be broken down by ball milling or pressing. This was achieved by further intelligent technologies by doping more effective elements and changing time of pre-heating and time of cooling the powder. Nine samples were prepared for study as solar laser materials as follows:



Figure-1. TEM scan of Nd: YAG solid state co-precipitation showing more soft samples and less agglomeration.



Figure-2. SEM of Nd: YAG Prepared by sol gel nitric acid co- precipitation method befor sintering.





Figure-3. Nd:YAG commercial powder mixture sol gel, no TEOS[sintered at 1750 ° C 20 hr].



Figure-4. TEM scan of Nd: YAG Sol gel Co-precipitation showing soft powder and spherical shape.



Figure-5. XRD pattern of Nd:YAG solid state Co-precipitation.

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Figure-6. XRD pattern of Nd:YAG sol gel Co-precipitation showing existence of single YAG crystal.



Figure-7. SEM morphology of Ce:YAG Mixed powder after calcination at 1100 °C 4 h.



Figure-8. Ce:YAG commercial powder mixture, no TEOS[sintered at 1750 ° C 20 hr].

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Figure-9. Ce:YAG commercial powder mixture, 0.5 % TEOS[sintered at 1750 ° C 20 hr] showing mono phase crystal highly compact.

Element	Weight %	Atomic %	Net Int.	Error %
C K	12.53	25.02	7.9	19.97
O K	31.33	46.96	63.1	12.2
AlK	20.91	18.59	268.7	5.71
Y L	34.54	9.32	193.98	3.95
CeL	0.69	0.12	1.78	62.77

Figure-10. EDAX of Ce YAG Mixed commercial powder method before sintering showing Y/Al% = 0.5

Figure-11. TEM image of Ce: YAG mixed commercial powder method showing particle size from 89 nm - 90nm.

Figure-12. SEM morphology of Ce: YAG sol gel co-precipitation after calcinations at 1100°C showing size of agglomerate 52 nm − 95 nm.

Figure-13. TEM morphology of Ce:YAG sol gel co-precipitation after calcinations at 110 °C showing spherical shape ranging from 10 nm -30 nm.

Figure-14. EDAX of Ce: YAG sol gel co-precipitation after calcination showing Y/Al% = 0.91%.

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Element	Weight %	Atomic %	Net Int.	Net Int. Error
AlK	24.56	51.91	508.4	0.01
Y L	74.17	47.57	640.1	0.01
CeL	1.26	0.51	4.9	0.56

Figure-15. XRD pattern of Ce:YAG Sol Gel Co-precipitated after calcination at 1100 °C 6h presenting the single YAG crystal formation.

Figure-16. IR spectra of Ce:YAG Sol Gel Co-precipitated with YAG peaks illustrated with other peaks discussed below.

Figure-17. SEM morphology of Ce:YAG powder precipitated in autoclave showing spherical Agglomerates that increases homogenous crystal formation, Before sintering.

Figure-18. SEM morphology of Ce:YAG precipitated in autoclave, 0.5 % TEOS [sintered at 1750 ° C 20 hr]

Figure-19. TEM image of Ce:YAG autoclaved showing elongated spherical agglomerates before sintering.

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Lsec: 81.9 0 Cnts 0.000 keV Det: Octane Pro Det Reso

Figure-20. EDAX of Ce : YAG autoclaved before sintering showing Y/Al%= 0.6.

Element	Weight %	Atomic %	Net Int.	Net Int. Error
AlK	33.18	62.06	156	0.01
Y L	66.65	37.83	117.4	0.02
CeL	0.03	0.01	0	0.76
NdL	0.05	0.02	0	0.95
CrK	0.09	0.09	0.2	0.8

Figure-20. (a) and Figure20(b). Showing Mapping of Nd and Ce homogeneous distribution over YAG area.

Figure-21. SEM morphology of Nd,Cr,Ce:YAG powder after calcinations and 1h autoclave showing fine powder and homogenous agglomerates.

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Figure-22. SEM morphology of Nd, Cr, Ce:YAG precipitated in autoclave 1 hr, 0.5% TEOS [sintered at 1750 °C 20 hr]showing big area of crystal compacted and mainly one phase.

Figure-23. TEM image of Nd,Cr,Ce:YAG powder after 1hautoclave and calcinations 6 h.

Figure-24. EDAX of Nd,Cr,Ce:YAG powder after autoclaved 1h and calcinations showing Y/Al% = 0.72.

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Element	Weight %	Atomic %	Net Int.	Error %
AlK	28.87	57.06	158.15	6.8
ΥL	69.96	41.95	151.54	6.13
CeL	0.13	0.05	0.13	99.99
NdL	0.18	0.07	0.17	99.99
CrK	0.85	0.87	1.82	70.99

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Figure-25. XRD pattern of Nd,Cr,Ce: YAG commercial powder mixture, autoclaved 1h and calcined at 1100 ° C 6 h illustrating sharp peak of single YAG crystal.

Element	Weight %	Atomic %	Net Int.	Error %
O K	41.1	66.56	90.64	11.27
AlK	24.36	23.39	388.77	7.02
Y L	33.15	9.66	243.44	5.34
CeL	0.48	0.09	3.09	61.21
NdL	0.48	0.09	2.94	61.42
CrK	0.43	0.21	5.94	43.33

Figure-28. SEM morphology of Nd, Cr, Ce:YAG crystal precipitated powder autoclaved 2h, no TEOS sintered at 1750 ° C 20h.

Element	Weight %	Atomic %	Net Int.	Error %
O K	42.57	68	96.43	10.9
AlK	23.53	22.28	380.98	6.89
Y L	32.91	9.46	248.51	5.1
CeL	0.36	0.06	2.31	66.21
NdL	0.36	0.06	2.23	63.4
CrK	0.28	0.14	3.95	62.2

Figure-29. EDAX of Nd, Cr, Ce:YAG crystal precipitated powder, autoclaved 2h and calcined at 1100 °C6 h before sintering, showing Y/Al % = 0.424.

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Figure-30. XRD of Nd,Cr, Ce:YAG Autoclaved 2h and calcined at 1100 °C6 h Prepared by sol gel method showing YAG formation with other phase.

UV Spectroscopic investigations:

Figure-31. Nd:YAG crystal solid state precipitation no TEOS , sintered 20h at 1750 °C showing the absorption peak of Nd around 300 nm with peak maxima 0.3.

Figure-32. Nd:YAG crystal commercial powder mixture no TEOS, sintered 20h at 1750 °C showing the absorption peak of Nd around 300 nm with peak maxima 0.3 too.

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Figure-34. UV absorbance peak of Ce:YAG crystal (commercial powder mixture) with 0.5% TEOS and sintered 20h at 1750 °C showing the absorption peak of Ce around 340 nm with peak maxima 0.25.

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Figure-38. UV absorbance peak of Nd,Cr,Ce:YAG sol gel autoclaved 2h at110 °C with 5% TEOS 20 h at 1750 °C showing broad absorption area with sharp peak around 300 nm with absorption maxima 0.1.

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Figure-39. TGA - DSC Curve of Nd,Cr,Ce:YAG crystal commercial powder mixture autoclaved 1h at 110 °C. Showing the powder thermal degradation.

Figure-40. TGA - DSC Curve of Nd,Cr,Ce:YAG crystal sol gel precipitation and autoclaved 2h at 110 °C. Indicating the crystal temperature formation.

3. DISCUSSIONS

Nd,Cr,Ce:YAG crystals were chosen to be prepared by doping neodymium and chromium and Ce ions. This is because energy is absorbed by the broad absorption bands of the Cr^{3+} doped and then transferred to Nd^{3+} by dipole - dipole interactions. The added cerium atoms strongly absorb in the ultraviolet region, and transfer their energy to the neodymium atoms. This material has been suggested for use in solar pumped laser, which could form part of a solar satellite system.

Nd,Cr,Ce:YAG crystals increases the pumping efficiency, the results is lower thermal distortion and high power output than Nd:YAG of the same pumping level. More over the lasing wave length, 1064 nm, is the same as for Nd:YAG. The material has a good resistance to damage caused by UV from the pump source, and low lasing threshold. Crystal of Yttrium Aluminum garnet doped with elements such as Nd, Ce, or Cr where Nd is often applied in steady state lasers emotions. Ce and Cr ions are used for increasing the crystal efficiency of arc lamp pumping. These ions takes up the greatest part of pump radiation and transfer it to generating ions. Another method that improves laser characteristics of different crystals is their annealing at high temperature (near melting point). During annealing process a partial removal of growth defects (e.g. oxygen vacancies) takes place. This method is time-consuming.

The chemical method of preparation used is the sol gel precipitation for most samples prepared in the laboratory. The samples were calcined at 1000°C, 1100°C for 2h, 4h - 6h in air.

3.1 The SEM morphology of sample after calcination and after sintering

3.1.1- Nd:YAG crystal precipitated with sol gel method and calcined at 1000°C 2h ,sintered at 1750°C 20h with no TEOS. Illustrated highly homogenous crystal with small number of holes and mainly one phase appeared. Pink in colour and opaque, Figure-2 and Figure-3.

3.2.1-Ce:YAG crystal precipitated by sol gel method, autoclaved and calcined at 1000°C 4h, sintered with 5% TEOS at 1750°C 20h. Illustrated moderately combined crystal, homogenous, with few number of pores and mainly two phases. The crystal was grey and opaque. Figure-7 and Figure-8.

3.3.1- Ce:YAG crystal prepared by commercial powder mixture and calcined at 1100 °C 4h, sintered at 1750°C 20h with 5% TEOS. Illustrated homogenous highly combined crystal with small number of different sized pores, translucent yellow in colour and showing mainly one phase two phases only, Figure-9.

3.4.1- Ce:YAG crystal commercial powder mixture, calcined at 1100°C 4h, sintered with no TEOS at 1750°C 20h. Illustrated homogenous crystal with three phases detected and large pores. The crystal was yellow in colour and opaque. Figure-12

3.5.1- Nd,Cr,Ce:YAG crystal precipitated with sol gel method and autoclaved 1h at 100-110°C, calcined at 1100°C 6h, sintered with 5% TEOS at 1750°C 20h. Illustrated homogenous big crystal aggregates, each crystal homogenous in content, showing major one phase, and minor two phase. The crystal was pink in colour and opaque. Figure-17

3.6.1- Nd,Cr,Ce,:YAG crystal precipitated by sol gel method using H_2O_2 , autoclaved at100-110°C 2h calcined at 1100°C 6h, sintered with no TEOS at 1750°C 20h. Illustrated homogenous big aggregate weak combined, with similar feature and major one phase. The crystal was pink in colour and opaque. Figure-18.

3.7.1- Nd,Cr,Ce:YAG crystal prepared by commercial mixture with acetone.

The mixture was calcined at 1100°C 6h, sintered with no TEOS at 1750°C 20 h. Illustrated high homogenous crystal, highly combined crystal with minor number of pores. The crystal was greenish with dark spots and opaque. Figure-21

3.8.1- Nd,Cr,Ce:YAG crystal prepared by commercial mixture with acetone.

The mixture was autoclaved 2h at 100°C, calcined at 1100°C 6h, sintered with 5% TEOS at 1750°C 20h. Illustrated highly combined crystal with high homogeneity with very small pores in size and number. The crystal was greenish and opaque, Figure-22.

3.9.1- Nd,Cr,Ce:YAG crystal prepared by commercial mixture with acetone. The mixture was autoclaved 2h at 100°C, calcined at 1100°C 6h, sintered with 5% TEOS at 1735°C 8h. Illustrated 5-10 microns grain size, two phases, pores up to10 microns. The crystal was taupe grey and opaque, Figure-26

3.10.1- Nd,Cr,Ce,:YAG crystal precipitated by sol gel method using, autoclaved at100-110°C 2h calcined at 1100°C 6h. Illustrated huge and weakly bounded grains (up to 100 microns). The crystal was reddish in color, Figure-28.

3.11.1- Ce:YAG crystal commercial powder mixture, calcined at $1100 \circ C$ 6h, sintered with 5% TEOS at $1700 \circ C$ 8h. Illustrated 5-10 microns grain size with homogenous Ce distribution, pores up to 5-7 microns large. The crystal was yellow in colour and translucent (one of the best crystals).

3.12.1- Ce:YAG crystal commercial powder mixture, calcined at $1100 \circ C$ 6h, sintered with 5% TEOS at $1735 \circ C$ 8h. Illustrated 5-10 microns grain size with homogenous Ce distribution, pores up to 5-7 microns large. The crystal was yellow in colour and translucent (one of the best crystals).

The SEM of doped-YAG with TEOS 0.5% and sintered at 1700°C - 1735°C and1750 °C for 8h and 20h respectively are shown in Figures (8-15-23-24-28). In spite of the different sintering temperature, ceramic obtained through adding TEOS sintering aid increased optical quality the whole sample is more homogenous and more compacted in crystallinity.

3.2 The TEM morphology of sample after calcination

TEM image shows high degree of crystallinity of selected region of the same sample confirmed by YAG aggregates in 89 - 90nm clusters that synthesized at 90 °C showing size of particles less than 100nm.

This may confirm the homogenous precipitation using autoclave. The shape of particles shows nearly spherical. The spherical particles would improve the accumulation density, which would influence the luminescent properties, Figure-1, Figure-4, Figure-11 Figure-13 and Figure-24.

Generally, the luminescent properties of particles depends on shape, defect and so on. The homogenous and spawn-like yttrium and aluminium precursor consisting fine particles aggregated with hydroxyls tightly, and forms the suspension as gel with rounded fibrillary structure. Thus, a small amount of sulphate ions effectively modifies the precursor morphology and influences the dispersion and the phase transmutation of the precursor and the phase translation of the precursor during the calcining process.

However sulphate ions adsorbed on the surface of the precursor hinder the translation of garnet phase and uniformed particle size of 89-90nmwere obtained.

3.3 The EDX results of Nd:YAG, Ce:YAG and Nd,Cr,Ce:YAG sample after calcination

The EDX results indicated that the particles contain both aluminium and yttrium, Nd, Ce and Cr elements. The results confirmed that aluminium compounds precipitated first in the form of nano dispersed spheres. With the decomposition of urea and the increase in the pH value, the yttrium compounds precipitated onto the surface of the existing sphere, making the smooth surface rougher.

Without sulphate ion the precursor is not spherical but gel-like, the ion may change the composition and surface energy of the nucleated Al³⁺ containing particles, and thus alter the crystal growth path way of precursor. The particle growth then follows the aggregation mechanism, in

which small particles aggregate into monodispersed spheres.

The EDS analysis of Ce:YAG granulated particle showed homogenous distribution Y/Al= 0.5 and 0.6 as expected in the raw suspension, very closing to theoretical value Y/Al=3/5=0.6. The Nd,Cr,Ce:YAG granulated particle showed homogeneous distribution Y/Al=0.7 and 0.42 for autoclaved 1h and autoclaved 2h respectively, Figure-10, Figure-14, Figure-20, Figure-24, Figure-27 and Figure-29.

This proved the perfect mixing of raw materials would short the diffusion distance between different elements and rapidly completed the densification of compact during sintering process. YAG phase is formed proving that all suspensions and granulated particles with different solid contents were homogeneous.

3.4 Mapping analysis

The sol - gel method, shows homogeneous distribution of Nd over YAG phase area. The Uniformity of the Ce distribution was investigated by mapping image too. Figure-(a) and (b) respectively.

3.5 The UV spectroscopic investigations after sintering at 1750 °C:

Samples of YAG doped with Nd ,ions or Ce, ions or Nd,Cr,Ce ions were pressed in diameters of 10-15mm and thickness 2 mm with both sides optically polished, were examined with UV radiation. All samples were investigated in the same conditions. Figures (31-38).

Displacement of short-wave edge of absorption was not found in any of the investigated materials, but there were found changes of the absorption coefficients near the edge, appearing inside rang of 200-800nm. It can be seen that among YAG crystals with different doped Nd:YAG, Ce:YAG, Nd,Cr,Ce:YAG shows significant sensitivity to UV radiation.

Figures (31-32) illustrating Nd:YAG crystals either precipitated with sol gel method or prepared by mixing with commercial YAG, both illustrated the same increase of the absorption maximum of 0.3.

Figures (33-34-35) Ce:YAG crystal prepared by sol gel method using autoclave and aided with 0.5%TEOS, then sintered 20h at 1750 °C shows highest increase in the absorption maximum of 0.5 absorbance. Both Ce:YAG crystals prepared by mixing Ce ions with commercial YAG powder and with no TEOS, sintered at 1750 °C, the maxima reached 0.35 absorbance only. The translucent Ce:YAG crystal which was sintered with 0.5%TEOS showed an absorbance maximum of 0.25.

The Nd,Cr, Ce:YAG crystal bands presented the Ce:YAG absorption peaks maximum at 340 - 400nm and additional peaks at 800-900nm. Figures (36-37-38).

3.6 The XRD Pattern of Nd: YAG and Ce: YAG Precursor and Nd, Cr, Ce: YAG crystals Prepared are discussed as follows:

Figure-5 and Figure-6 illustrates the XRD pattern of Nd:YAG solid state preparation and Nd:YAG sol gel preparation. For comparison, the difference between the peak position of the solid state preparation and the sol gel method may be due to the lower temperature in preparation and type of additives. It should be noted that the brightness of the prepared particles is due to preparation conditions, Furthermore, the effects of precursor, urea and sulphate affects size and morphology of produced particles.

Figure-15 shows the XRD pattern of Ce:YAG crystal prepared by sol gel technique indicated complete phase transformation to YAG structure.

Figure-25 and Figure-30 illustrates the XRD patterns of doped YAG by Nd,Cr,Ce ions, either by mixing for commercial powder or by sol gel precipitation respectively. Figure-25 illustrated complete phase transformation to YAG structure for autoclaved powder 1h and calcined at $1100 \,^{\circ}$ C for 6 h. Figure-30 illustrated reduce in the full width of half-maximum of crystallinity and appearance of other phases when the autoclave time was increased up to 2h. This result may be attributed to H₂O₂ and ozone gas used besides more time of autoclave.

3.7 The TGA-DSC analysis for prepared Nd, Cr, Ce: YAG precursor

The TGA-DSC analysis of commercial powder mixture of precursor where the endothermic peak occurred around 111 °C results from evaporation of absorbed water and the release of molecular water. The exothermic peak around 156 °C mainly attributed to the removal of organics. The endothermic peaks from 350 °C - 450 °Care due to the decomposition of residual CO₂, NO₂ and NH₄ in the powder as shown in Figure-39.

The TGA-DSC analysis of sol gel precipitated precursor illustrates the overall mass losses which occurred below 400°C is due to evaporation of absorbed water, peak at 97 °C, and the decomposition of nitrate, peak at 176°C may be attributed to organic substances combustion. Final decomposition stage of Nd,Cr,Ce:YAG crystal is continuous up to 650 °C - 800 °C. It is clear that the crystallization peak temperature of YAG is 750 °C as shown in Figure-40.

CONCLUSIONS

Most optical materials have been wide optical gap, monolithic and dense glasses (oxides, fluorides glasses, etc) semiconductors and single crystals. But ceramics, made of wide gap materials can increase the panoply of optical materials for the reason of the simpler fabrication and better thermos-mechanical and chemical resistance.

- Nd:YAG powder were successfully fabricated by sol gel method and solid state method. The SEM of sol gel method shows much better homogeneity and after calcination at 1000 °C 2h. Sintering with TEOS 0.5% proved that vacuum sintering at 1750 °C 8-20h improved crystal formation.
- Ce:YAG powders were successfully produced by either mixing with commercial YAG or by sol-gel coprecipitation. Both when calcined at 1100 °C 6h. Meanwhile 2 molar % of Ce³⁺ doping concentration has been proved good for emission intensity of samples as shown in UV- absorption figures and TEM figures.

- Ce:YAG fabricated with mixing method and calcined up to 1100°C 6h ,developed the sample doped 2% atCe³⁺ to appear translucent when sintered 20 h at 1750°C 20h.
- A highly homogenous ion-doped yttrium aluminium garnet nano power synthesized with[U]/[m] ratio by the urea homogeneous precipitation method in presence of sulphate ions were formed and transformation during calcination which agglomeration during calcination due to addition of the appropriate amount of sulphate ions.
- Nd,Cr.Ce:YAG nano powder with uniform size and good dispersity were synthesized by co-precipitation at low temperature with autoclave1,2h at110°C and a follow up calcination 6h at 1100°C
- This evenly resulted in the formation of homogenous Co-doped Nd,Cr,Ce:YAG with good dispersity and uniform sizes. The ion:YAG nano powders with uniform particle size 67 - 89 nm were obtained at 1100°C 2-6h.
- XRD for powders of Nd,Cr,Ce,:YAG prepared by autoclave1h showed better crystalline structure than that prepared by autoclave 2h and using ozone gas.
- Mainly all powders showed good UV- absorption spectra for Nd:YAG, Ce:YAG, or mixed elements such as Nd,Cr,Ce:YAG.
- The above facts suggest that sol gel YAG powder is the most effective and easiest for evaluating functional bulk YAG optical materials expecting for evaluation and design of optical properties not only YAG but also of other oxides such as Nd,Cr,Ce doped ions.
- Microstructure analysis revealed that lowering the sintering temperature, showed samples with larger grained matrix with clusters of small grains. TEOS is sintering aid which was used successfully in Italy to sinter samples at 1700 °C 8h, 1730 °C 8h, and 1750 °C 20 h.
- Results showed that samples sintered at 1750 °C 20 h were fully dens with less or minor number of pores and no other defects were found between or in grains.

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