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Review paper

Production of nanopowder and bulk aluminate ceramic scintillators

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Abstract

The developed nanotechnology makes it possible to prepare lots of standard powders of Aluminates such as lutetium and yttrium orthorombic $LuAlO_3$, $YAlO_3$ (LuAP, YAP – perovskite), aluminum garnets $Lu_3Al_5O_{12}$, $Y_3Al_5O_{12}$ (LuAG, YAG) with cubic structure and aluminates with monoclinic structure $Lu_4Al_2O_9$, $Y_4Al_2O_9$, (LuAM, YAM). Powders of scintillation materials are prepared by sol-gel method, co-precipitation method and a combination of sol-gel method with combustion reactions. Crystallization process of LuAG phase starts at 900 °C. At lower temperatures, during the pyrolysis of the gel, amorphous powders have been produced. After pyrolysis of the gels without additives of aminoacetic acid a mixture of LuAG + Lu_2O_3 has been formed instead of LuAP. New phase of LuAP in a cubic form is detected. Diffraction peak profile analyzes and scanning electron microscope (SEM) investigations are performed to approximately establish grain size of the obtained powder. Thermogravimetry analysis (TGA) was carried out for detection of thermal effects in the range up to sintering temperature. Perovskite phases of aluminates have been synthesized via pyrolysis (at <900 °C) of the gel. Prepared nanopowders of aluminates are sintered using spark plasma processing. The best density of YAG has been detected at the following conditions of SPS process: 1650 °C/3 min/40 MPa.

Keywords: Aluminate scintillators; Sol-gel syntheses; Spark plasma sintering

Contents

1. Introduction

Aluminates represent one of the widely used aluminum-based composite materials. Among aluminates one of the most utilized materials are lutetium-aluminates used as single crystal scintillators, manufactured by Chokhralsky method [1]. It is a rather complex, long-standing and power-intensive process. Therefore, their cost is high and an area of application is very restricted. Elimination of the costly and time-consuming crystal growth process using optically transparent ceramics or glasses offers a promising approach to fabrication of relatively inexpensive ceramic scintillators [2]. For optical applications, ultra-fine powders with particle size of several to several hundreds of nanometers are needed. Generally, powder metallurgy methods are widely applied to production

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of aluminates. Three different complex-oxides are known to be stable crystalline phases in Al_2O_3–Y_2O_3 system: YAM (yttrium aluminum monoclinic $Y_4Al_2O_9$), YAP (yttrium aluminum perovskite $YAlO_3$) and YAG (yttrium aluminum garnet $Y_3Al_5O_{12}$) [3,4]. The polycrystalline powders of those oxides are commonly synthesized by solid-state reaction between yttrium and alumina at temperatures above 1600 °C. However, increase in temperature often made it difficult to prepare very fine powders, which are important for manufacturing materials of optical applications. In addition, obtaining each individual single phase is not that easy, because of simultaneous formation of the above oxides due to inhomogeneous chemical composition, complicated phase transitions and decomposition of Al_2O_3–Y_2O_3 system at high temperature. On the other hand, though, recent remarkable advances in chemical procedures, such as the sol-gel process and other related techniques using organic chemicals, prove that those oxides, especially YAG, can be synthesized at much lower temperatures. YAP is one of the stable crystalline complex oxides and has been known as a promising optical material. However, it is difficult to get fine single-phase powders using conventional high-temperature solid-state reactions, due to agglomeration and formation of other phases such as YAG and YAM [3–6]. Detailed study of Nd doped YAG was conduct by E. Caponetti and M. L. Saladino. From the quantitative XRD investigation, by the Rietveld method, formation of a single phase cubic garnet structure (YAG:Nd 5–6 at.%) by co-precipitation method is attained at temperatures as low as 900 °C [7–11].

Pure phase lutetium aluminum garnet (LuAG) powders have been obtained by Y. K. Liao. The paper describes co-precipitation method mixing solution of aluminum and lutetium nitrate using ammonium hydrogen carbonate (AHC) as precipitant, followed by calcination of the dried precipitates at 1000 °C. The author claims that this is pivotal for the control of chemical homogeneity within the particles as well as for agglomeration and size of the particles. Finally, the synthesized powder is produced as a single-phase LuAG with the weakly agglomerated fine spherical grains (20 nm average) and a relatively narrow grain size distribution [1]. In the meantime H.L. Li in a publication describes production process of nanosized LuAG:Ce powder by the sol-gel combustion method. The purified crystalline phase of LuAG:Ce is obtained at 820 °C directly crystallizing from amorphous materials. According to this work maximum luminescent intensity of LuAG:Ce can be reached at Ce concentration of 0.5 at.%. In addition, the luminescent intensity can be increased with increasing the calcining temperature due to the improved crystallization [12].

Preparation of ceramic scintillators requires compaction and consolidation (sintering) of the initial nanocrystalline powders. Densification of polycrystalline ceramics to optical transparency in the visible range is yet a challenge and requires prolonged sintering time at very high temperatures under high vacuum conditions. The sintering process itself results in an increase in particle size leading to a loss of transparency due to the particle size surpassing the wavelength of light. As to the ceramic scintillators, it has been generally accepted earlier that transparent polycrystalline ceramics would be limited to those materials exhibiting a cubic crystal structure. Given that non-cubic structures are characterized by birefringence effects in the randomly oriented grains, this leads to multiple scattering of light and either translucency or total opacity. Recently, spark plasma sintering (SPS) is used for rapid densification of translucent and transparent functional and nanocrystalline ceramics. [13,14].

In this study, we applied sol-gel and co-precipitation methods for establishing possibilities of synthesis of three Y–Al–O compounds, and discussed the results with an emphasis on the temperature dependence of their phases.

2. Experimental

Synthesis of lutetium aluminates (LuAG) is possible using soluble compounds of aluminum ($Al(OR)_3$, $Al(NO_3)_3 \cdot 9H_2O$, $Al_2(SO_4)_3 \cdot 18H_2O$, $AlCl_3$, etc.). LuAG can be obtained through sol-gel procedure (co-precipitation method) as well as through pyrolysis of the gels obtained by evaporation of salt solutions. At the same time, preparation of LuAP is rather complex process. Phase purity of aluminates depends on the nature of components of sol-gel process and on the conditions of the gel pyrolysis. Lutetium oxide (purity 99.9%), cerium nitrate (purity 99.9%), aluminum nitrate (purity 99.9%), aluminum (purity 99.9%), 96% C_2H_5OH, 97% formamide, 56% HNO_3, 24% NH_4OH and 37% HCl were used as starting materials. Other reagents were of an analytical range.

Powders of scintillation materials were prepared by sol-gel method, co-precipitation method and a combination of sol-gel method with combustion reactions. A Nanocrystalline scintillation material of lutetium aluminate (LuAG) was obtained in our experiments using the following procedure:

1. Lu_2O_3 (1.2 g) was dissolved in nitric acid (56% mass, 10 ml). The obtained solution was evaporated up to the consistence of syrup, and diluted with ethyl alcohol (90% vol, 50 ml). Solution of aluminum nitrate (3.8 g $Al(NO_3)_3 \times 10\ H_2O$, 50 ml) was prepared separately and to this solution were added 2 ml of dilution of cerium nitrate (III) (4 g $Ce(NO_3)_3 \times 10\ H_2O$,10 ml H_2O) and 5 ml of glycine. The solutions was combined into one and agitated for 10 min. Then 20 ml of ammonia 12% mass solution was gradually added to the solution. The obtained sol was heated upon agitating and held in this condition for 2 h; then the reaction mass was transferred to the porcelain cup for evaporation. The obtained gel was heated for 1 h up to 200 °C resulting in a powder of yellowish color, subsequently transferred into quartz crucible and burned at 900 °C for 1 h on the air or in the inert gas (Ar, N_2). The resulted product was white with a tint of yellow.
2. Preparation of LuAG by co-precipitation: solution of cerium nitrate (2 ml) and 0.05 mol aluminum nitrate

(50 ml) were added to 0.03 mol of lutetium nitrate (50 ml of water). Metal hydroxides were precipitated from the solution of ammonia (20 ml 12% mass) upon agitating. Reaction mass was agitated at room temperature for 3 h and then filtered. Sediment was washed with dissoluted aqueous solution of ammonia (3×50 ml; 0.5–1% (mass)) and dried in air flow (~ 40 °C). The obtained gel-like mass was burned in a muffle furnace upon gradual rising of temperature up to 900 °C. The obtained powder was held at this temperature for 1–3 h.

3. Preparation of fine dispersion lutetium aluminate: 0.05 mol aluminum nitrate (50 ml) was added to 0.03 mol of lutetium nitrate and diluted in 10 ml of water with adding 100 ml ethyl alcohol, solution of cerium nitrate (2 ml) and 0.05 g glycine. During agitation, the solution was evaporated up to obtaining syrup-like mass. Then the obtained mass was carefully dried at 100–130 °C (30 min.). The obtained brownish mass was dispersed and put into a preliminarily heated (up to 700 °C) tubular furnace. Reaction proceeds rapidly (1–3 min), giving a voluminous powder ($\sim$80–120 ml) as a result. Temperature of the furnace was increased up to 900 °C and was held for 1 h. Then the powder was cooled in the air and stored in the exsiccator.
4. The synthesis of nanocrystalline LuAP was realized using organic additives by following way: solutions of 0.003 mol lutetium (III) nitrate and 0.003 mol aluminum (III) nitrate were mixed and 1 ml of nitric acid (56%) and 0.5 g aminoacetic acid were added. The resulted solution was evaporated. The formed gel was powdered and heated in a furnace up to 700 °C. Reaction was finished in about 1–3 min. The formed powder (80–120 ml) was calcinated at 900 °C for 1 h. Color of the powder (LuAP) was yellowish-white.

   LuAP has been synthesized through pyrolysis (at < 900 °C) of the gel obtained from $Lu(NO_3)_3$–$Al(NO_3)_3$–NH_4OH–NH_2CH_2COOH system. The same gel at > 900 °C creates several phases:

   $Lu(OH)_3 + Al(OH)_3 \rightarrow LuAP$ (< 900 °C)

   $Lu(OH)_3 + Al(OH)_3 \rightarrow$
   $LuAP + LuAG + Lu_2O_3$ (1000 °C)

   $Lu(OH)_3 + Al(OH)_3 \rightarrow$
   $LuAG + LuAP + LuAl_3O_9$ (1200 °C)

5. Pure nanocrystalline powder of YAG was formed due to sol-gel process. Yttrium oxide (Y_2O_3) nitrate of aluminum ($Al(NO_3)_3 \times 9H_2O$) and cerium oxide (CeO_3) were used as starting materials. Precursors were dissolved in the nitric acid and after evaporation DEAM (Dietanolamin) was added to provide nanocrystallinity of the powder. Drying was carried out at 150 °C during 30 min and synthesis at 900 °C for 1 h.

Briefly, experimental procedures for producing powder of different aluminate materials are given in Table 1.

The nanopowders of aluminates were sintered via SPS in graphite environment at 7–10 V and high-frequency current of 2000–2750 A for 2–8 min under 20–40 MPa. To avoid carbon penetrating into the lattice structure of molybdenum, thin films were used. However, some kind of molybdenum and carbon oxide also exhibited interaction with sample. Therefore, after compaction annealing was performed in air at 1450 °C for 10 h.

Metallic content in the reagents was determined by the atomic-absorption method (Perkin-Elmer atomic-absorption spectrometer Analist 600). The analyses of organic compounds were carried out by Agilent Technologies GC/MS methods (6890 N/5975).

Crystalline phases of the powders were identified by X-ray diffraction method (XRD) using XZG-4 diffractometer with CuKα radiation ($\lambda = 1.5418$ Å). For approximate determination of grain size of the obtained powder, diffraction peak profile analyses were carried out in addition to scanning electron microscopy (SEM) study. Thermogravimetry analysis (TGA) was carried out by means of Setsys Evolution (Setaram Tag 24). The carbon content in gels and powders was analyzed by the Multi EA 2000 analyzer. After appropriate measuring of scintillation properties, powders had been consolidated and sintered by SPS. Temperature dependence of nanocrystallinity and density were established. After investigation of structural conditions of bulk pieces by XRD and Scanning Electron

Table 1
Experimental procedures for producing powder of different aluminate materials.

#	Material	Method	Precursors	Preparation of charge (Temp./Time)	Syntheses of powder (Temp./Time)
1	LuAG	Sol-gel	Lu_2O_3, $Al(NO_3)_3 \times 10H_2O$ $Ce(NO_3)_3 \times 10H_2O$ Glycine	200 °C/1 h	900 °C/1 h
2	LuAG	Co-precipitation	$Lu(NO_3)_3 \times 10H_2O$ $Al(NO_3)_3 \times 10H_2O$ $Ce(NO_3)_3 \times 10H_2O$	Room temp./3 h	900 °C/1–3 h
3	LuAG	Sol-gel method + combustion reactions	$Lu(NO_3)_3 \times 10H_2O$ $Al(NO_3)_3 \times 10$ H_2O $Ce(NO_3)_3 \times 10$ H_2O Glycine	100–130 °C/30 min, 700 °C/1–3 min	900 °C/1 h
4	LuAP	Sol-gel method + combustion reactions	$Lu(NO_3)_3 \times 10$ H_2O $Al(NO_3)_3 \times 10$ H_2O	700 °C/1–3 min	900 °C/1 h
5	YAG	Sol-gel method	Y_2O_3, $Al(NO_3)_3 \times 10H_2O$ CeO_3, DEAM	150 °C/30 min	900 °C/1 h

Microscope (SEM) the best working conditions for SPS process were determined.

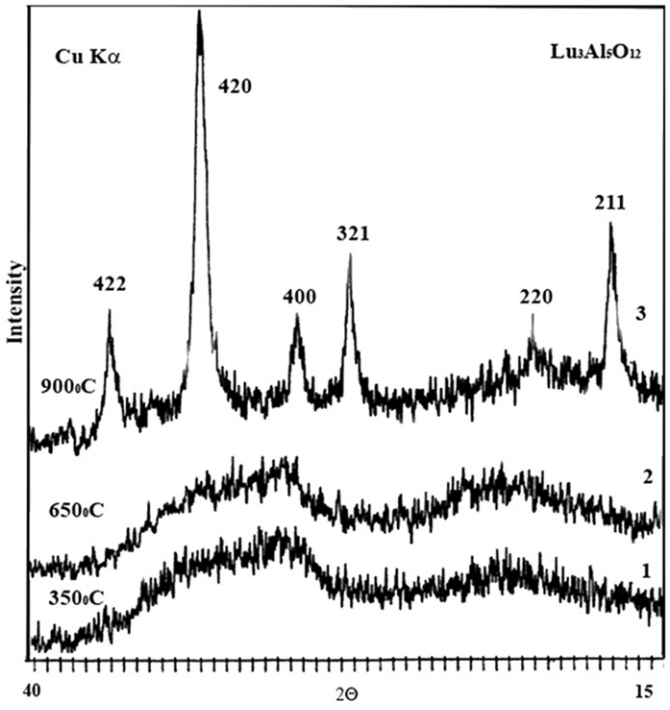

Fig. 1. X-ray diffraction patterns of the powders of lutetium aluminate LuAG synthesized at temperatures: 1–350 °C; 2–650 °C; 3–900 °C (PDF #73-1368).

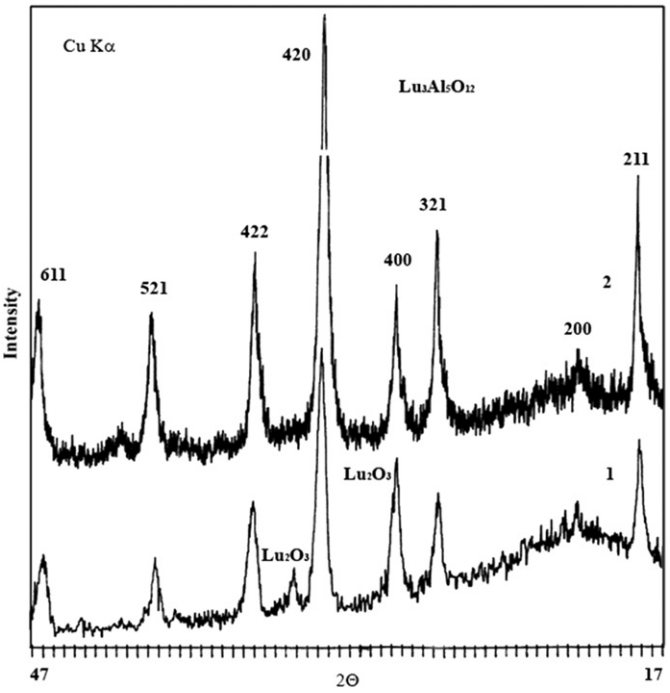

Fig. 2. X-ray diffraction patterns of the two-phase (1) and single-phase (2) powders of lutetium aluminate.

3. Results and discussion

Crystallization process of the LuAG phase starts at 900 °C. At lower temperatures (> 900 °C) amorphous powders are produced. After pyrolysis of the gels without additives of aminoacetic acid, mixture of LuAG + Lu_2O_3 was formed instead of formation of LuAP. Fig. 1 shows

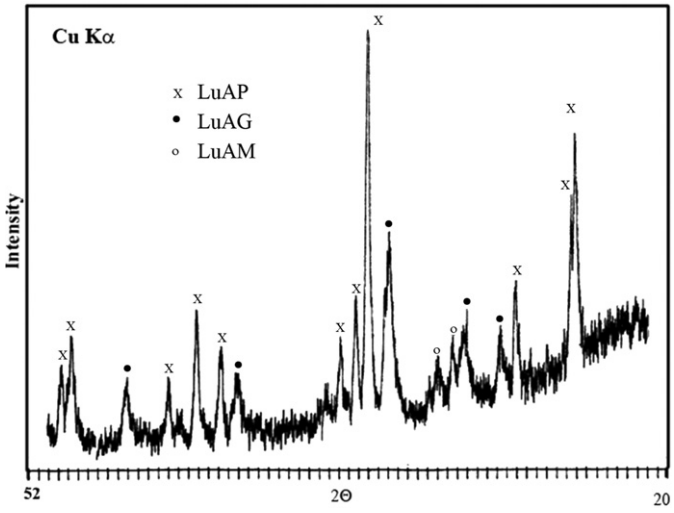

Fig. 3. X-ray diffraction patterns of the powders of synthesized LuAP (PDF# 24-0690) with the other phase LuAG and LuAM (PDF# 33-0844).

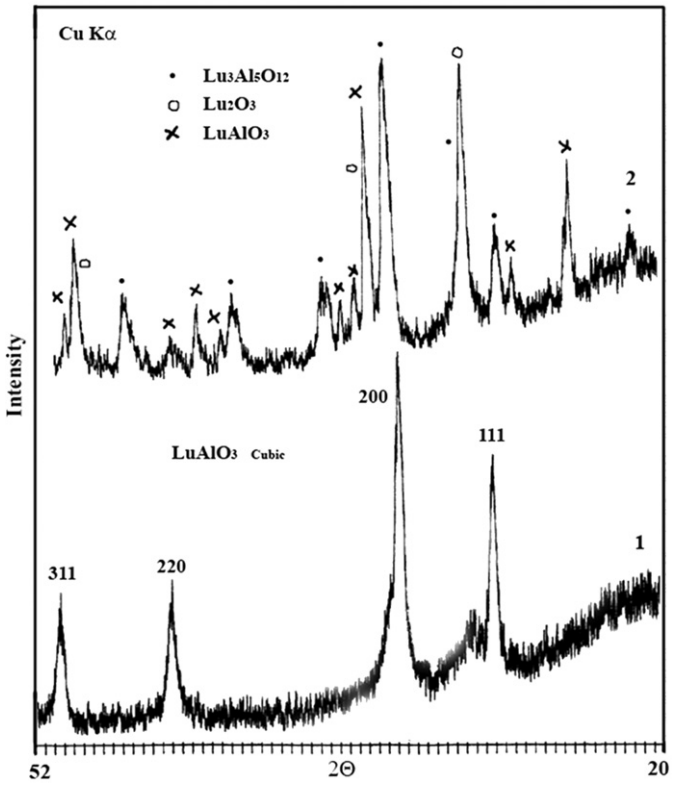

Fig. 4. X-ray diffraction patterns of the powders of synthesized LuAP: (1) cubic form; (2) phases of transformation.

x-ray diffraction patterns of the powders of lutetium aluminate LuAG synthesized at different temperatures.

The experiments showed that lutetium aluminates are synthesized in roentgen amorphous state at temperatures up to 700 °C. Nanocrystals start forming at 750 °C and nanocrystalline structure is completely formed at 900 °C. Deviations from the strict technological modes provide formation of lutetium oxide as a second phase that is shown in the figure (Fig. 2, x-ray pattern 1). The same figure shows single-phase structure of lutetium aluminates (x-ray pattern 2). Increase in temperature of LuAG synthesis up to 900 °C does not change structural state, however, sizes of crystallites increase. Despite the fact that main goal of our experiments was fabrication of cubic lutetium aluminates (LuAG), we had attempts to fabricate powders of LuAP as well. As shown from the results of our experiments the latter can be obtained, though, its formation is accompanied with the formation of other phases, as LuAG and LuAM (Fig. 3).

To our opinion, if the experiments continue it will be possible to obtain single-phase powder of LuAP. As far as we are concerned, such kind of phase does not exist at low temperatures. Besides the expected orthorhombic phases, revealed in our experiments on fabricating LuAP, we have frequently observed formation of LuAM, and to our opinion, of an additional new phase of LuAP in a cubic form. During heating above the synthesis temperature, this new cubic phase transforms into two phases – orthorhombic LuAP and lutetium aluminate LuAG, where formation of lutetium oxide is also observed (Fig. 4, diffraction pattern 2). Yttrium aluminate YAP with orthorhombic and hexagonal structures has been obtained as a structural modification of LuAP (Fig. 5).

Our supposition on the formation of lutetium aluminate LuAP and on its transformation is supported with a single reaction:

$$5\mathrm{LuAP} \rightarrow \mathrm{LuAG} + \mathrm{Lu_2O_3}\ (1000\ ^\circ\mathrm{C})$$

$$7\mathrm{LuAP} \rightarrow \mathrm{LuAG} + \mathrm{LuAM}\ (1100\ ^\circ\mathrm{C})$$

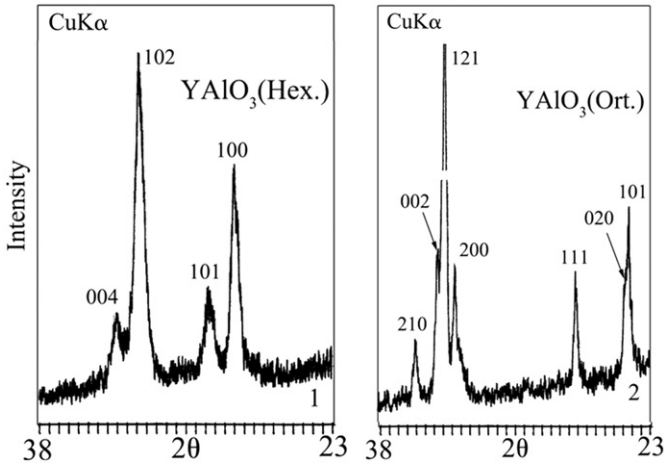

Fig. 5. X-ray diffraction patterns of the powders of synthesized YAP: (1) Hexagonal structure (PDF# 55-0621). (2) Orthorhombic structure (PDF# 33-0041).

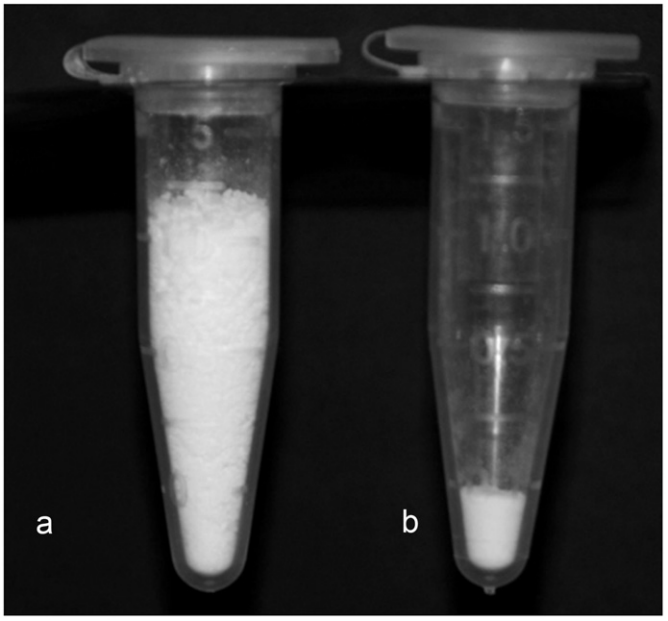

Fig. 7. Volume comparison of (a) nanopowder and (b) coarse powder of YAG.

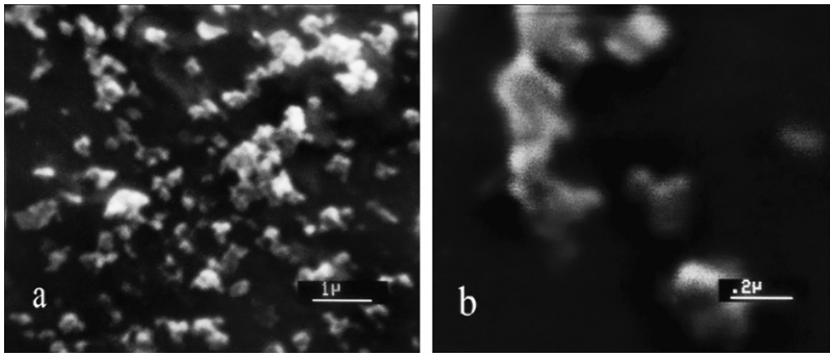

Fig. 6. SEM micrographs of nanocrystalline YAG powder.

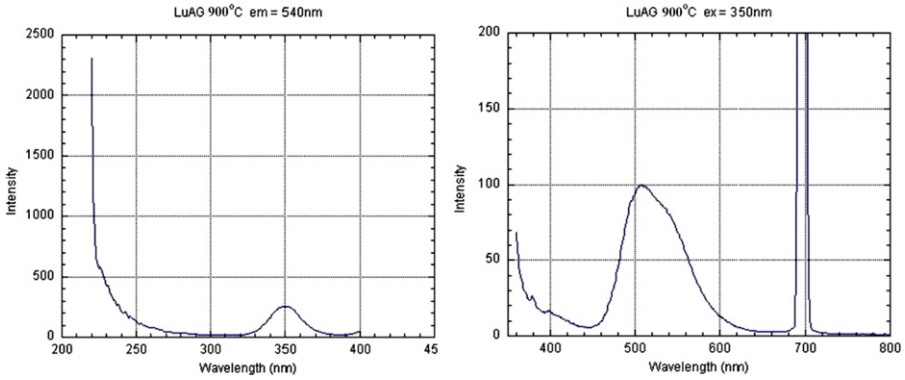

Fig. 8. Excitation and emission spectra of nanocrystalline LuAG powder synthesized at 850 °C.

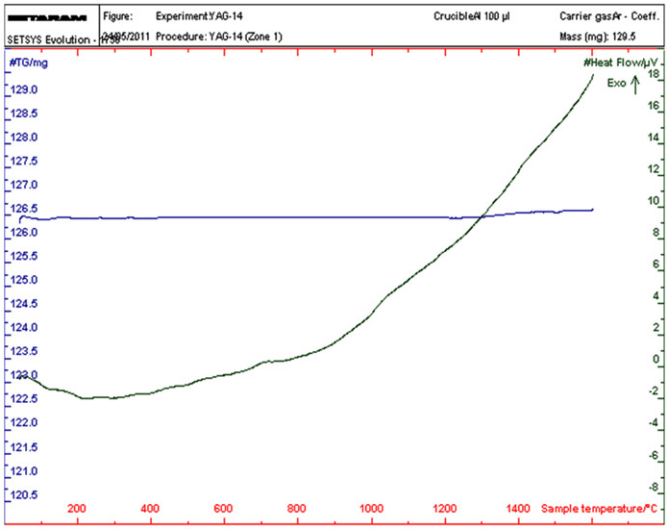

Fig. 9. TG and DTA curves of YAG nanopowder.

Diffraction peak profile analyses were performed to establish approximate grain size of the obtained powder. According to the Scherrer formula crystallite size (L) could be calculated from the following equation:

$$L_{cr} = K\,\lambda/\mathrm{FWHM}\cos\Theta$$

where K is the constant for cubic crystals is equal to 0.94; λ, the wavelength of the X-ray used (0.154 nm); FWHM, the width of the diffraction peak measured in radians; Θ the Bragg angle of the peak.

According to the calculations of the YAG powder, grain size is approximately less than 100 nm. In this equation, we did not take into consideration broadening due to instrumental and lattice strain effects. Obviously, results could not describe exact crystallite size. Nevertheless, it gives us possibility to estimate approximate grain size of the obtained powder. SEM micrographs prove nanocrystalline structure of alumina powders with slight agglomerations (Fig. 6).

Volume of nanocrystalline powder considerably exceeds volume of the powder obtained using ordinary technology (Fig. 7).

Crystalline structure (within the limits of nanocrystallinity) of the powder of aluminate lutetium LuAG with garnet structure is very well formed, though it was synthesized at a considerably low temperature (900 °C). Despite of this fact, intensity of excitation and emission spectra (Fig. 8) is extremely low. On the excitation spectrum (within the range of 200–450 nm) which is in correspondence with emission with the wavelength 540 nm, one well defined maximum is observed corresponding to the wavelength of 350 nm. Emission spectrum of the material has been taken at excitation radiation of 520 nm. One can see that the maximum of emission spectrum with low intensity is in correspondence with the radiation of 520 nm wavelength while the maximum with better intensity corresponds to 700 nm.

Thermal analysis of the obtained YAG nanopowders showed that the amount of organic contaminations included in the material could not be easily removed by a low-temperature heating. As an example, the results of thermal analysis of the YAG nanopowders are shown in Fig. 9. No changes were observed for Mass reduction or Endothermic effect till 1600 °C.

After appropriate measuring of scintillation properties, powders have been consolidated and sintered by SPS. Results shown in Table 2, indicate impact of technological regimes on the characteristics of the samples.

As it is obvious from the SEM images (Fig. 10), after sintering of nanopowder at (1100 °C:2 min:35 MPa), sample remains weakly sintered and requires higher temperature to achieve better density. Also, it was found that absolute elimination of porosity is essential to achieve complete transparency. Consequently, increase in sintering temperature and pressure is necessary.

Results of the experiments showed that for achieving transparency, graininess of the starting powder is not significant due to the cubic structure of YAG. For reduction of porosity and improvement of sintering quality, an ultrasound excitation will be used during SPS process.

Table 2
SPS operating modes with relatively of density transparency.

Sample No.	SPS current (V/A)	Temp. (°C)	Holding time (min)	Pressure (Mpa)	Density (% of theoretical)	Remarks
SPS-164$_{(Nano)}$	7.5/2100	1100	2	35	94.7	Opaque
SPS-172$_{(Nano)}$	8/2500	1500	8	20	97.5	Translucen
SPS-272$_{(Nano)}$	9.5/2700	1600	3	20	97.4	Translucen
SPS-284$_{(Coarse)}$	9/2600	1550	3	20	96.8	Opaque
SPS-320$_{(Coarse)}$	10/2300	1650	3	40	98.1	Semitransparente
SPS-325$_{(Coarse)}$	9/2500	1600	8	30	97.6	Translucen

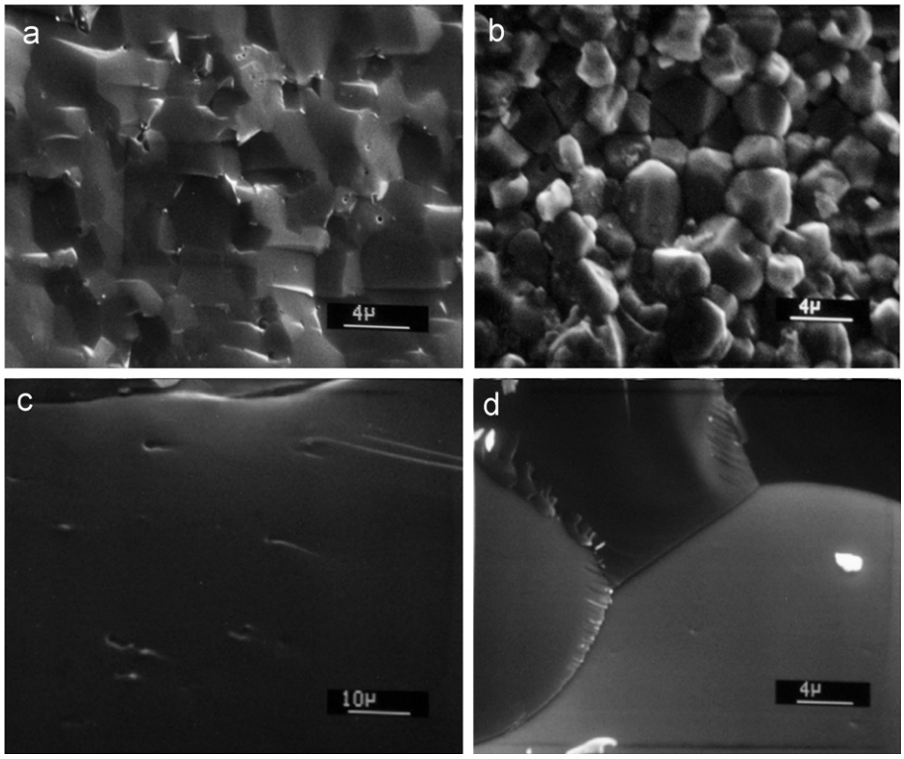

Fig.10. YAG sintered at the SPS different mode obtained from (a) nanopowder at 1600 °C:3 min:20 MPa; (b) nanopowder at 1100 °C:2 min:35 MPa; (c,d) coarse powder at 1650 °C:3 min:40 MPa.

4. Conclusion

Pure nanocrystalline powder of aluminates has been prepared by sol-gel method, co-precipitation method and a combination of sol-gel method with combustion reactions.

It is difficult to preserve phase purity in $(Y,Lu)_2O_3$–Al_2O_3 system. Application of different additives and solvents provides influence on formation of nanocrystalline pure powder. New method makes it possible to obtain monophase powder of LuAP. Such kind of phase does not exist at low temperatures. Besides the expected orthorhombic phases in the experiments on fabricating of LuAP, we frequently observed formation of LuAM and of an additional new phase of $LuAlO_3$ in a cubic form. During heating above the synthesis temperature this new cubic phase transforms into two phases – orthorhombic LuAP and lutetium-aluminates LuAG, where formation of lutetium oxide is also observed. The experiments showed that the nanocrystalline structural state and the single-phase structure condition of the synthesized lutetium-aluminates are completely formed at 900 °C. The best density of YAG was detected at the following conditions of SPS process: 1650 °C/3 min/40 MPa. Further work will be directed to the detection of ultrasound excitation influence during sintering process, and aluminates with perovskite and monoclinic structure will be sintered via new method. Still, the main challenge is to preserve nanocrystalline structure of bulk samples that ensures transparency.

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