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Synthesis of nanopowders by the glycine-nitrate method in the In-Dy-O system

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We investigated the feasibility of synthesizing nanopowders containing indium and dysprosium oxides by the glycine-nitrate method. It was found that the glycine-nitrate method significantly reduces the content of the indium component in the resulting mixture of indium and dysprosium oxides. In this case, intensive absorption of carbon dioxide from the air by the formed particles induces formation of amorphous carbonaceous compounds, which decompose only under high-temperature treatment (>900 °C). This prevents compaction of powders synthesized by the glycine-nitrate method. Comparison of the characteristics of powders containing indium oxides and dysprosium, synthesized by the glycine-nitrate method and by the method of co-precipitation of indium and dysprosium hydroxides from chloride solutions, showed the advantage of the co-precipitation method in the pressing of powders.

Keywords

glycine-nitrate method nanopowders indium oxide dysprosium oxide hydroxide co-precipitation method

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Key findings

- The feasibility of synthesizing nanopowders containing indium and dysprosium oxides by the glycine-nitrate method is shown.
- The glycine-nitrate method significantly reduces the content of the indium component in the resulting mixture of indium and dysprosium oxides.
- The absorption of carbon dioxide from the air by particles formed during the glycine-nitrate synthesis induces formation of amorphous carbonaceous compounds (probably, basic dysprosium carbonates) in the synthesized powders; these carbonaceous compounds when decomposed, prevent compaction of the powders obtained by the glycine-nitrate method.
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1. Introduction

Oxide targets for magnetron sputtering are currently in great demand among the ceramic materials based on oxides of rare and rare earth metals [1]. Powders containing oxides of indium and dysprosium are used in the manufacture of ceramic targets for deposition of the corresponding films for sensors [2] and optoelectronics [3, 4]. The fabrication of these targets requires powders of high dispersion (0.1–0.05 μm) and purity without pre-grinding. Reduced size of the powders allows producing ceramic materials of a submicron structure and high density [5]. The most well-known methods for

obtaining such nanopowders are co-precipitation [6], hydrothermal synthesis [7, 8], sol-gel technology [9], laser ablation [10], and direct deposition [11].

One of the most effective methods for the synthesis of oxide nanopowders is the method based on precipitation of hydroxides from a mixture of metal salts with an alkaline agent [12]. This method does not require expensive equipment; it is technologically advanced and can be easily scaled up. As shown in [13], the deposition method can be used for production of powders to manufacture targets for deposition of oxide films containing indium and dysprosium. A significant disadvantage of the method of

chemical precipitation of metal hydroxides is a high degree of agglomeration of the synthesized powders and their wide grain-size distribution [14]. In order to increase the dispersion of the final powders (by reducing the coagulation of deposited particles and preventing agglomerate formation), the technique of solution combustion synthesis has been developed in recent years [15-26]. The process involves an exothermic reaction between an oxidizing agent such as metal nitrates and a fuel such as urea (H2NCONH2), carbohydrazide (CO(NHNH2)2), or glycine (C₂H₅NO₂). The combustion reaction is initiated at temperatures of the order of 500 °C and below. In a typical reaction, the precursor (a mixture of water, metal nitrates, and fuel) decomposes, dehydrates, and ignites upon heating. The reaction yields a bulky product (foamy powder) that occupies the entire volume of the reaction vessel. The synthesized nanomaterials are generally homogeneous, contain fewer amounts of impurities, and have a larger surface area compared to powders produced by conventional solid phase methods. The amount of starting substances is evaluated based on calculations of redox reactions. The solution combustion synthesis method shows many potential advantages such as low cost, energy efficiency, and high productivity [15, 20].

The most widely used version of the solution combustion method is the glycine-nitrate method [27]. Synthesis of a complex oxide by the glycine-nitrate method is performed as follows [28]. Solutions of metal nitrates are taken in stoichiometric amounts appropriate for synthesis of a complex oxide or a solid solution and mixed with glycine. The mixture is evaporated. After evaporation of excess water, the reaction mixture turns into a homogeneous liquid of syrupy consistency. Upon further heating, the mixture spontaneously ignites, and the combustion induces the formation of oxide particles. Self-ignition occurs at temperatures Ts ranging from 150 to 900 °C, while the Ts value depends on the composition of the synthesized oxide. The reaction proceeds quickly and violently. The process runs in a self-sustaining mode and ends when the fuel runs out completely. The flame temperature varies from 1100 to 1450 °C depending on the glycine-nitrate ratio. The resulting loose and very light 'ash' contains the reaction product, usually oxides, and soot. The gaseous reaction products are carbon dioxide, nitrogen, and water vapor.

Numerous studies address the glycine-nitrate synthesis of chromites, cobaltites, ferrites, gallates, cuprites, manganites, and other complex oxides as well as solid solutions based on them. Among these, few papers report on the synthesis of indium oxide doped with tin [29] and dysprosium oxide [30, 31]; however, the data on the glycine-nitrate method used for producing powders of mixed indium and dysprosium oxides are not available in the literature.

In this work, we studied the feasibility of obtaining powders of the In-Dy-O system by the glycine-nitrate method for the production of ceramic targets.

2. Experimental

Compositions based on nanocrystals of indium and dysprosium oxides were obtained by glycine-nitrate combustion. Indium (III) and dysprosium (III) nitrates, $In(NO_3)_3 \cdot 4.5H_2O$ and $Dy(NO_3)_3 \cdot 5H_2O$ (reagent grade, Plant of rare metals, Russia), and glycine H_2NCH_2COOH (pure, pharma grade, Panreac), taken in a stoichiometric ratio, were used as initial reagents at a molar ratio of Dy:In = 1:1. The equation for the reaction can be represented as follows:

$$3Dy(NO_3)_3+3In(NO_3)_3+10C_2H_5NO_2\rightarrow 1.5Dy_2O_3+$$
 $1.5In_2O_3+14N_2+20CO_2+25H_2O$
(1)

The equation indicates a stoichiometric course of the reaction ensured by the ratio of glycine and nitrates in the mixture G/N = 0.56.

The solution of nitrates of dysprosium, indium, and glycine was evaporated (90–100 $^{\circ}$ C) in a tube furnace (Figure 1) to a gel-like state, and then the temperature was raised to ~300 $^{\circ}$ C. The process was accompanied by spontaneous ignition of the mixture with a rapid release of gaseous reaction products, an increase in the volume, and the formation of nanopowders. The product of glycine-nitrate synthesis is a foamy substance. Additional heat treatment of nanopowders was carried out in air at a temperature of 1000 $^{\circ}$ C for 1 h.

After that, the resulting powders were studied by thermal and X-ray diffraction analyses. Thermal analysis was performed using an STA 449 F1 Jupiter simultaneous thermal analyzer coupled with a QMS 403 Aeolos mass spectrometer (NETZSCH-Geraetebau GmbH, Germany) in the temperature range of 30–600 $^{\rm o}$ C at a heating rate of 10 $^{\rm o}$ C min $^{-1}$ in the ambient atmosphere. The measurements were performed in corundum crucibles.

Phase composition of the powders was determined by X-ray diffraction analysis (XRD) using an XRD-6000 (Shimadzu) diffractometer in Cu K α radiation (Tomsk Materials Research Multiple Access Center NR TSU).

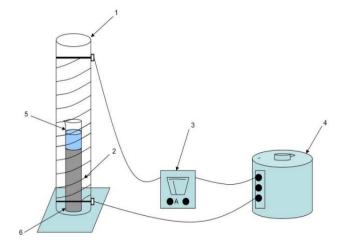


Figure 1 Schematic drawing of the set-up for glycine-nitrate synthesis of nanopowders: (1) quartz tube, (2) heating coil, (3) ammeter, (4) autotransformer, (5) quartz glass filled with the solution; (6) porcelain bases [32].

The scanning range was $10-80^{\circ}$ (20), the scanning step - 0.02°, and the acquisition time was 1 s. The diffraction patterns were identified with PDF-cards No. 04-003-4703 and 00-006-0416 (International Centre for Diffraction Data). Structure refinement as well as phase ratio and lattice parameter calculations were performed by Rietveld Method (full-profile analysis) with PowderCell2.4 free software. IR spectra of the samples were registered by an IR Fourier spectrometer Nicolet 6700 equipped with a Smart Orbit™ diamond ATR accessory (Thermo Scientific, USA) using a single-beam optical scheme and a built-in Michelson interferometer. The spectra were obtained within the range of 400-4000 cm⁻¹ with resolution of 4 cm⁻¹. The specific surface was studied using a 3Flex adsorption analyzer (Micromeritics, USA) with respect to the data on nitrogen adsorption at -196 °C. The specific surface area (Ssp) was determined with a relative error of $\Delta \pm 10\%$ using the multipoint (10-12 points) Brunauer-Emmett-Teller (BET) method at relative nitrogen pressure P/P_0 ranging from 0.05 to 0.30.

Prior to measuring the specific surface area, the samples were degassed (at 150 °C in vacuum) for 2 h. The surface morphology was studied using a Philips SEM 515 scanning electron microscope equipped with a combined electron and focused ion beam system Quanta 200.

The powder particle size was determined using a SALD-7101 nano particle size analyzer (SHIMADZU, Japan). The principle of its operation is based on static laser light scattering with light wavelength λ = 375 nm (laser diffraction method). An aqueous solution of sodium citrate with a concentration of 0.2 mass.% was used as a dispersion medium.

The samples were prepared by uniaxial static pressing in the form of pellets with a thickness of 3–6 mm and a diameter of 9 mm. The compaction kinetics of the ceramic target was studied using a highly sensitive horizontal dilatometer DIL 402 C (NETZSCH, Germany).

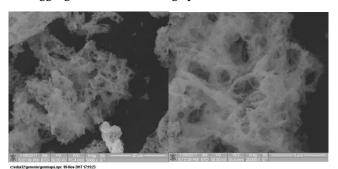
3. Results and Discussion

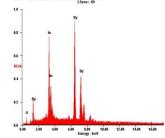
Figures 2 and 3 present the electronic images and the results of the quantitative analysis of powders of the In-Dy-O system, which were synthesized by the glycine-nitrate method after thermal treatment and without it. The powders consist of indium, dysprosium, oxygen, and are loose aggregates with a developed surface. During heat treatment, they are compacted. Quantitative analysis indicates a significant loss of indium in the composition of the synthesized powder. This is also confirmed by the results of the XRD analysis (Figures 4, Table 1). The powders contain the Dy₂O₃ and In₂O₃ phases, while the Dy₂O₃ particles are larger than the In₂O₃ particles, and the particle sizes increase during heat treatment. At the same time, the content of the In₂O₃ phase is more than three times lower than that of Dy₂O₃. Heat treatment of powders at 1000 °C does not change the phase ratio. This is probably due to the high temperatures of the glycine-nitrate method

(more than 1000 °C). It is known that when heated above 1000 °C In_2O_3 partially dissociates with the formation of volatile In_2O :

$$In_2O_3 = In_2O + O_2.$$
 (2)

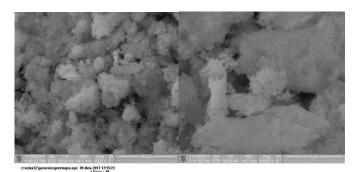
The curves of the quantitative distribution of powder particles (a typical curve is presented in Figure 5) show that the particle size of the powder synthesized by the glycine-nitrate method varies from 0.353 to 1.622 μm , and the measured specific surface area of the powder ranges from 11.54 to 15.46 m^2/g . This indicates that the Shimadzu SALD-7101 3D particle size analyzer still detects aggregates formed during synthesis.

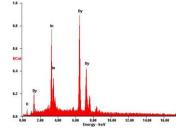




Element	Wt.%	At.%
OK	03.41	27.08
InL	30.81	30.03
DyL	65.78	42.89
Matrix	Cor-	ZAF

Figure 2 Electronic images and results of the quantitative analysis of the powders of the In-Dy-O system synthesized by the glycine-nitrate method at a molar ratio of Dy:In = 1:1 without additional thermal treatment.





Element	Wt.%	At.%
OK	03.99	27.08
InL	31.78	30.03
DyL	64.23	42.89
Matrix	Correc-	ZAF

Figure 3 Electronic images and results of the quantitative analysis of the powders of the Dy-In-O system synthesized by the glycine-nitrate method at a molar ratio of Dy: In=1:1 with additional thermal treatment at 1000 °C for 1 h.

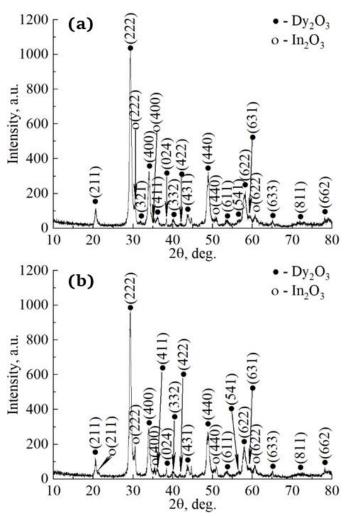


Figure 4 X-ray pattern of the obtained powders of the In-Dy-O system synthesized by the glycine-nitrate method at a molar ratio of Dy:In = 1:1 without additional thermal treatment (a) and 1 with additional thermal treatment at 1000 $^{\circ}$ C for 1 h (b).

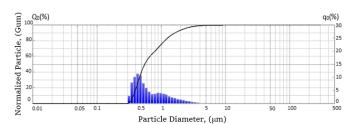


Figure 5 The curve of quantitative distribution of the powder particles synthesized by the glycine-nitrate method.

As reported in [5], powders that consist of aggregates with strong interparticle bonds are not promising for the synthesis of ceramics, since the intraaggregate cavities not eliminated by pressing turn into pores during subsequent sintering, which shrink only at very high temperatures. Moreover, during sintering, agglomerates with loose particle packing form large pores, which shrink only after the long-term exposure at high temperatures.

Figure 6 shows DTA, DSC, and TG curves (a) and mass thermogram (b) characteristics of the studied powders. These curves indicate the release of H₂O and CO₂ from the powders at elevated temperatures (>900 °C). IR - spectroscopy of the nanopowders indicates the presence of several intense absorption bands, the main of which are the result of the sorption of gaseous combustion products, such as H_2O (3470 cm⁻¹), CO (2270 cm⁻¹) and CO_2 (1467–1526, 1386–1413, 1050 cm⁻¹). It can be assumed that self-ignition of the glycine-nitrate mixture during synthesis initiates intensive absorbtion of carbon dioxide from the air by the formed particles. As a result, amorphous carbonaceous compounds are formed, which decompose at temperatures >900 °C. This process hinders the compaction of powders synthesized by the glycine-nitrate method, as evidenced by the dilatometric curves in Figure 7 (curves 1 and 2).

4. Limitations

We demonstrated the feasibility of synthesizing the nanopowders containing indium and dysprosium oxides by the glycine-nitrate method. It was found that absorption of carbon dioxide from the air by particles formed by the glycine-nitrate method induces formation of amorphous carbonaceous compounds in the powders. These compounds decompose and hinder the compaction of powders synthesized by the glycine-nitrate method. Confirming this conclusion requires further additional experiments involving IR spectroscopy, X-ray phase and thermal analyses. In this regard, it is essential to further study the powder properties after their disaggregation and degassing by any method. A changed ratio of glycine to nitrates (G/N) and the temperature of thermal treatment of powders can be used as variable parameters to reduce the loss of the indium component in the composition of powders.

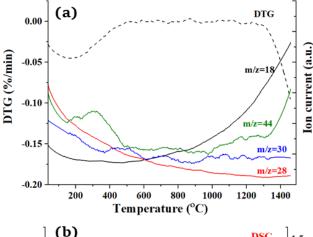
Table 1 Results of the XRD analysis of the powders obtained by glycine-nitrate method.

Sample*	Detected phases	Phase content, mass.%	Lattice parameters, Å	Deviation from card data **, Δ	CSR size, nm	$\Delta d/d \cdot 10^{-3}$
Dy:In-1:1,	Dy ₂ O ₃ _206	79	a = 10.5700	0.1	37	5.4
without thermal treat- ment	In ₂ O ₃ _206	21	a = 10.2100	0.092	19	8.3
Dy:In-1:1,	Dy ₂ O ₃ _206	78	a = 10.5700	0.1	50	6.1
thermal treatment at 1000 °C for 1 h	In ₂ O ₃ _206	22	a = 10.2100	0.092	37	8.4

^{* –} with the indicated composition and thermal treatment after citrate sol-gel synthesis.

 $I/I_{\rm cor}$ (In₂O₃) = 9.3 (10.118 Å, #6-416); 13.22 (10.140 Å, #22-336); 13.02 (10.117 Å, #71-2194); 13.03 (10.1192 Å, #89-4595). $I/I_{\rm cor}$ (Dy₂O₃) = 8.3 (10.665 Å, # 22-612); 11.54 (10.665 Å, #43-1006); 12.77 (10.630 Å, # 79-1722); 12.76 (10.6706 Å, #86-1327).

^{** -} for In_2O_3 _206 a = 10.118; for Dy_2O_3 _206 a = 10.6700.



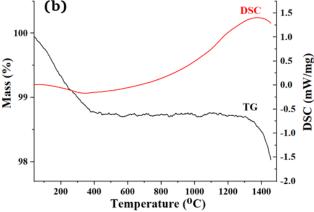


Figure 6 DTA, DSC and TG curves (a) and mass thermogram (b) for the powders of the In-Dy-O system synthesized by the glycine-nitrate method (molar ratio of Dy (III):In (III) = 1:1). $m/z = 28 - NO_2$, m/z = 30 - NO, $m/z = 18 - H_2O$, $m/z = 44 - CO_2$.

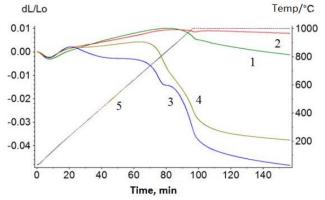


Figure 7 Dilatometric curves of the powders of the In-Dy-O system at compaction pressures $P=775\,$ MPa (curves 1 and 3) and $P=155\,$ MPa (curves 2 and 4). Powders 1 and 2 were synthesized by the glycine-nitrate method, powders 3 and 4 were obtained by the hydroxide co-precipitation method.

5. Conclusions

The study showed the feasibility of synthesizing the nanopowders containing indium and dysprosium oxides by the glycine-nitrate method.

It was found that the glycine-nitrate method is not feasible as a technological stage of powder formation for ceramics due to the loss of the indium component and intensive absorption of carbon dioxide from the air by the formed particles, which is accompanied by the formation of amorphous carbonaceous compounds that decompose at high temperatures.

Comparison of the characteristics of powders containing indium and dysprosium oxides, which were synthesized by the glycine-nitrate method and by co-precipitation of indium and dysprosium hydroxides from chloride solutions, showed the advantage of co-precipitation method in pressing of powders.

• Supplementary materials

No supplementary materials are available.

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Author contributions

Conceptualization: T.D.M. Data curation: G.S.A. Formal Analysis: A.I.A. Funding acquisition: V.V.Z.

Investigation: G.S.A., A.I.A., V.V.Z.

Methodology: T.D.M.

Project administration: G.S.A.

Resources: V.V.Z. Software: A.I.A. Supervision: T.D.M. Validation: G.S.A. Visualization: V.V.Z.

Writing - original draft: T.D.M.

Writing - review & editing: T.D.M., G.S.A.

Conflict of interest

The authors declare no conflict of interest.

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