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Comparison of dispersants performance on the suspension $Lu_2O_3:Eu^{3+}$ stability and high-density compacts on their basis

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Abstract

The conditions for obtaining a stable $Lu_2O_3:Eu^{3+}$ suspension of spherical particles with a diameter of 100 nm using three dispersants possessing an electrosteric stabilizing effect (Dolapix CE 64, Darvan 821 A, Darvan C-N) have been studied. It has been shown that in colloidal processing of ceramics the packing density and microstructure of green bodies can be controlled by regulating the interactions between ceramic particles in the suspension. The influence of the molecular weight and concentration of the dispersant on the stability of $Lu_2O_3:Eu^{3+}$ suspensions containing 5–10 vol.% of the solid loading has been considered. It has been determined that use of Dolapix CE 64 with a concentration of 1 mass.% in the alkaline pH range allows to obtain suspensions with high stability and low viscosity (~1.7 cP). Such suspensions were used to produce compacts with a maximum relative density of ~52% and uniform density distribution by the pressure slip casting method. The obtained compacts were densified into translucent $Lu_2O_3:Eu^{3+}$ ceramics by the vacuum sintering method.

Keywords: A. Slip casting; Slurry; Lu_2O_3; Dispersant

1. Introduction

Compaction is one of the most critical stages in the optical ceramics technology aiming at producing of high density green bodies with homogenous microstructures. However, it can lead to undesirable defects, such as pores, and to non-uniform density distribution in the obtained samples. To a considerable extent, the formation of defects depends on characteristics of the initial powders, as well on the compaction method applied. The use of nanopowders is a necessary condition for obtaining transparent ceramics because it allows to decrease the temperature of vacuum sintering in comparison with that for the micron-sized powders and, consequently, to obtain fine-grained ceramics [1,2]. ''Dry'' pressing methods (uniaxial and isostatic) do not provide uniform density distribution in the samples, due to interparticle friction, since in this case sliding and rearrangement of particles are strongly limited, thus decreasing the density of the compact [3,4]. To provide more homogeneous packing of the nanoparticles during the molding process and make high-density compacts, ''wet'' compacting methods are preferred. Such methods based on self-organization of the powder particles are alternative for ''dry'' pressing. One of them is the pressure slip casting (or pressure filtration) [5,6]. As this method uses a liquid phase, it permits to considerably diminish interparticle friction forces. Moreover, due to the action of capillary forces, homogeneous compacts with a packing density of about 55–62% can be obtained [5–7]. However, the use of nanopowder as a starting material essentially impedes the compaction process. Due to their high reactivity caused by excess of free surface energy, nanoparticles tend to agglomerate under the influence of Van der Waals and adhesive forces that lead to the formation of skeleton structure in the green bodies which hinders powder densification [6]. To achieve close packing of nanoparticles in a compact by the slip casting method, it is necessary to obtain a stable concentrated suspension with a high solid loading of 10–30 vol.%. Such a suspension must possess the Newtonian liquid nature, i.e. provide independent motion of each particle. Stability of the

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slip is achieved by the introduction of a suitable dispersant into the suspension. Thereat, it is very important to provide uniform distribution of dispersant over the surface of the particles. Typically, to obtain a stable suspension on the basis of nanoparticles it is necessary to introduce not less 0.2 mg of dispersant by square meter of powder specific surface [8]. Preparation of stable suspensions from Y_2O_3 and YAG powders using Poly(acrylic) acid (PAA), Dolapix CE 64, Dispex A40, Darvan C-N, Duramax D3021, etc. as dispersants was reported in [9–12]. However, there is practically no data on the preparation of Lu_2O_3 suspensions in the recent literature, though Lu_2O_3 ceramics doped with rare-earth ions are promising materials for solid-state lasers, optical elements and X-ray detectors [13,14]. To our best knowledge, only in [14] the use of slip casting for lutetium oxide molding is mentioned. Compared to other rare earths oxides such as Y_2O_3 (ρ = 4.84 g/cm^3), Lu_2O_3 possesses a very high density (9.424 g/cm^3), therefore it is difficult to prepare a stable suspension, since the particles tend to extremely fast sedimentation due to gravity. This problem can be solved by the choice of an appropriate dispersant and its optimum concentration. The obtaining of suspension with suitable rheological properties is a significant task in the colloidal processing of ceramics such as slip casting, since this allows one to control the consolidation process and the resulting compact density.

The goal of the present work was to study the influence of the type, molecular mass and concentration of the commercial dispersants Dolapix CE 64, Darvan 821 A and Darvan C-N on the stability of Lu_2O_3:Eu^{3+} suspensions and the resulting density of the compacts obtained by slip casting.

2. Experimental procedures

2.1. Starting materials

The suspensions were obtained from Lu_2O_3:Eu^{3+} powder consisting of spherical particles with an average diameter of ~100 nm (the dispersion of the size was less than 10%) and a specific surface area of 20 m^2/g measured by the BET method. The powder was prepared by the homogeneous co-precipitation method with subsequent calcinations of the precursor at 800°C during 2 h according to [15]. The commercial available polyelectrolytes Dolapix CE 64 (Zschimmer and Schwarz GmbH, Germany), Darvan 821 A and Darvan C-N (R.T. Vanderbilt Company, Inc., USA) were used as dispersants. These dispersants are the derivatives of carboxylic acid and contain –COO^-–NH_3^+ group. A detailed characterization of the dispersants is presented in Table 1.

2.2. Slurry preparation and slip casting

The Lu_2O_3:Eu^{3+} suspensions containing 5, 10 vol.% (14.2 g, 37.7 g, respectively) of the solid loading were prepared by mixing of lutetium oxide, bidistilled water and dispersants (0.5–1.5 wt.%). The dispersant concentration was calculated with respect to the mass of Lu_2O_3:Eu^{3+} powder. The mixture was submitted to homogenization in a polyethylene container during 20–40 h by ball milling using polyamide balls. The mass ratio of the grinding bodies and of the powder was 4:1. To remove air bubbles formed during homogenization, the slip was treated under vacuum during 10–20 min. The pH value of the slip was maintained within the range from 9 to 10 using ammonia water solution (NH_4OH). The suspension stability was estimated by sedimentation tests performed for the suspensions with different dispersants. At first, the suspensions were kept in 20 cm^3 cylinders for 24 h, and then their stability was determined from the weight of sediment. The sediment has been separated from suspension, dried up at room temperature and weighed. The prepared suspensions were used to obtain compacts by the slip casting method. The slurry was placed into metallic mould with a punch of 10 mm in diameter and passed through a membrane filter with an average pore size of 100–200 nm. The dispersion liquid was absorbed by porous Al_2O_3 ceramics. The pressure was ranged between 65 and 150 MPa. The compacts obtained from consolidated Lu_2O_3:Eu^{3+} powders were shaped as disks with a diameter of 10 mm and a thickness of 1–2 mm. After drying in air at room temperature, the green bodies were annealed at 1200 °C in oxygen flow with a rate of 2 l/h to remove the organic components. Then the compacts were sintered for 8 h in vacuum of 10^{-3} Pa and a temperature of 1800 °C.

2.3. Characterization techniques

The size of the powder particles, the morphology and microstructure of the obtained samples were characterized by scanning electron microscopy (SEM) using JSM-6390 LV (JEOL, Japan) and transmission electron microscopy (TEM) using EM-125 (Selmi, Ukraine). The viscosity of lutetium oxide suspension was measured by an Ostvald viscometer with a capillary diameter of 1.12 mm. The measurements were carried out at 25 °C. The density of the compacts was measured by the hydrostatic weighing method. Thermal analysis (DTA-TG) was realized using a derivatograph MOM Q-1500 in air in the 20–1000 °C temperature range, the heating rate was 10 °C min^{-1}. The α-aluminum oxide was used as a primary standard. Vacuum sintering of the samples was performed in a

Table 1
Properties of aqueous solutions of the dispersants used.

Dispersant	Dolapix CE 64	Darvan 821 A	Darvan C-N
Nomenclature	Polymethacrylate, ammonium salt	Polyacrylate, ammonium salt	Polymethacrylate, ammonium salt
Molecular weight (g/mol)	350	3500	10,000–16,000
Active matter (wt.%)	70%	40%	25%
Density (g/cm^3)	1.10	1.16	1.10–1.12

SNVE-1.3.1/20 I2 furnace with tungsten mesh heating elements (VNIIETO, Moscow).

3. Results and discussion

3.1. Suspension stability

The morphology of the starting Lu_2O_3:Eu^{3+} powders used to prepare the slurry for slip casting is shown in Fig. 1. The synthesized powders meet the main requirements for the preparation of transparent ceramics, i.e. small particle size (of the order of 100 nm), spherical shape and monodispersity [5,16].

Macromolecules of Dolapix CE 64, Darvan 821 A and Darvan C-N polyelectrolytes contain the ionogen $–COONH_4$ groups which form the negatively charged carboxylate polyions (COO^-) and the ammonium ions during hydrolyzation in alkaline media [17]. In an aqueous media lutetium oxide particles tend to surface hydration, thus forming a certain quantity of $Lu(OH)_3$. As lutetium hydroxide is a weak base, it dissociates insignificantly, $k_{dis} = 10^{-24}$ (the least for lanthanide series). $Lu(OH)_3$ dissociation is stepwise and results in the formation of the LuO^+ cation:

$$Lu(OH)_3 \leftrightarrow Lu(OH)_2{}^+ + OH^- \quad (1)$$

$$Lu(OH)_2{}^+ \leftrightarrow LuO^+ + H_2O \quad (2)$$

According to the Paneth–Fajans–Hahn rule, the macrocation LuO^+ is adsorbed on the surface of Lu_2O_3, since it contains lutetium ion (a component of the oxide crystal lattice), therefore the particle acquires a positive charge. We assume the following structure of lutetium oxide micella in aqueous medium:

$$\{m[Lu_2O_3]nLuO^+(n-x)OH^-\}^{x+}xOH^- \quad (3)$$

The nature of the interaction between the negatively charged dispersant ion and the positively charged surface of Lu_2O_3 particle may be explained within the Stern theory. The dispersant polyions are incorporated into the adsorption layer of the particle due to strong electrostatic interactions and high adsorptivity caused by polarization. Owing to high charge density, the polyions not only neutralize positive charge of the solid surface, but also recharge the particle negatively by changing the sign and value of its ζ-potential. The value of the created ζ-potential defines the main slurry parameters – stabilization efficiency and fluidity. The higher the absolute value of this potential is, the more stable suspension is formed. The ζ-potential of the suspension can be also influenced by regulating its pH value, because hydrogen and hydroxyl ions possess high ability to be adsorbed. The former have a small radius, and can be closely spaced with respect to the surface of the solid phase, the latter have a high dipole moment. Therefore, in most cases there are observed two regions of maximum fluidity and maximum ζ-potential values of opposite sign. One of these regions is located in acid medium; the other is in alkaline medium. The ζ-potential value and its dependence on pH for Lu_2O_3 suspensions are absent in the literature. However, Y_2O_3 is a structural analogue of Lu_2O_3, one can conclude that the behavior of the electrokinetic potential of Lu_2O_3 will be similar to that of Y_2O_3 suspensions [18]. This means that the maximum suspension stabilization is achieved by additive influence of the dispersant and the suspension pH. The synergetic action of a large number of –OH groups and large dispersant polyions in alkaline medium increases the ζ-potential of the particles. The most stable Lu_2O_3:Eu^{3+} slurry was obtained at pH ∼ 9–10, because increase of the ζ-potential gives rise to deflocculation of the particles thus creating preconditions for the slurry thinning. At the said pH values polyacrylic and polymethacrylic acids completely dissociate, demonstrating maximal stabilizing effect [19]. In the intermediate region (pH 4–6) where recharge of the particles takes place, the ζ-potential value diminishes and goes to zero at a certain point, whereas the suspension viscosity considerably increases.

So, the suspension stabilization process is realized under the influence of a number of factors. The increase of the surface charge gives rise to electrostatic repulsion of the particles (electrostatic effect). Long carbon chains of the dispersant molecules surrounding the particles create their additional spatial separation (steric effect). The combination of the electrostatic and steric mechanisms gives rise to electrosteric stabilization of the disperse system typical for polyelectrolytes. Moreover, water molecules orientated around the monomolecular layer of the adsorbed dispersant, form a hydrate shell. Thereat, the surface tension forces at the liquid-solid interface weaken, additionally enhancing aggregation stability of the suspension.

The model of the dispersant adsorption on the particle surface is shown in Fig. 2. Due to repulsion of the closely spaced groups with the same charge, the polyions occupy much

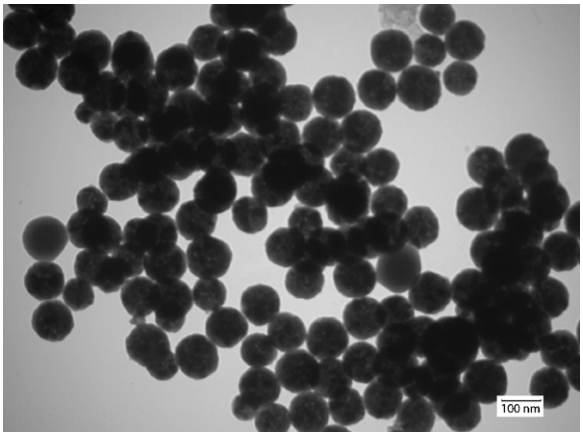

Fig. 1. TEM image of Lu_2O_3:Eu^{3+} powders.

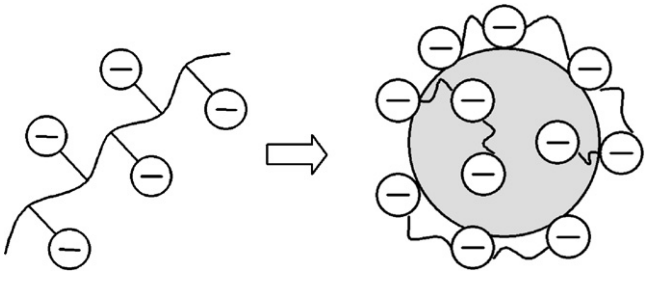

Fig. 2. Model of dispersant adsorption on the surface of Lu_2O_3:Eu^{3+} particle.

larger volume than the corresponding electrically neutral macromolecules. When being adsorbed, the dispersant wraps a particle owing to the presence of a large number of polar groups.

3.2. The slurry homogenization

To homogenize the slurry, i.e. to achieve uniform distribution of the dispersant over surface of lutetium oxide, ball milling was realized for 20 or 40 h. The distribution of the particles in the slurry after drying is presented in Fig. 3. The water contained in the slurry facilitates destruction of ''soft'' agglomerates, since it penetrates into surface microcracks and exerts a wedging effect. During the homogenization ''soft'' agglomerates are being destroyed, the dispersant covers the particles and prevents the formation of new ones. The decrease of the process efficiency with time (more than 40 h) is defined by a number of factors, first of all, by the distribution of the dispersant over the surface of each of the particles and by their spatial separation, which is clearly seen in the picture (sample 3).

3.3. Effects of dispersant type and concentration on stability of Lu_2O_3:Eu^{3+} aqueous suspension

To obtain stable concentrated slurry with 5, 10 vol.% of lutetium oxide, the sedimentation and aggregation stability of homogeneous aqueous suspensions based on spherical monodispersed Lu_2O_3:Eu^{3+} nanoparticles stabilized by different dispersants (Dolapix CE 64, Darvan 821 A and Darvan C-N) were studied. The quality of the slurry (its stability) was estimated from the mass of the sediment formed in the suspension after 24 h. The data obtained were used to optimize the type and concentration of the dispersant, i.e. to provide the highest suspension stability and the lowest sediment content. As can be seen in Fig. 4, Dolapix CE 64 is the optimal dispersant. At a low dispersant concentration (lower than 1 wt.%) the repulsive forces of the nanoparticles are insignificant, agglomeration in the liquid phase still remains, so that the suspension rapidly settles (curve 1). In general, low dispersant dosage is not sufficient to completely deflocculate the suspension and maintain the colloidal particles in dispersion. At the dispersant content of ∼1 wt.% the polyelectrolyte

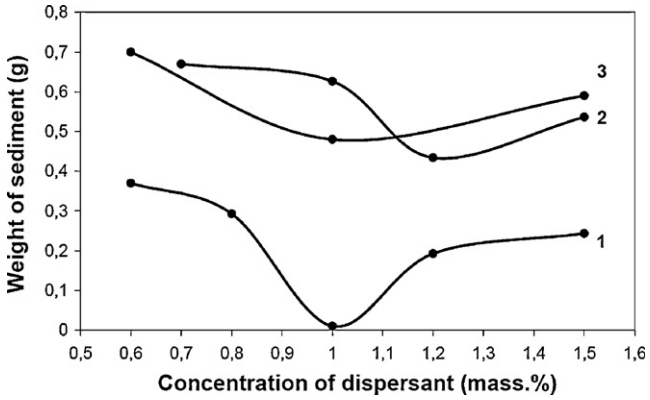

Fig. 4. The sediment weight as a function of dispersant concentration: 1, Dolapix CE 64; 2, Darvan 821 A; and 3, Darvan C-N.

molecules wrap each particle, thus providing the balance between the attractive and repulsive forces. Such a suspension is most suitable for slip casting. However, further increasing of dispersant concentration (up to 1.2 wt.%) leads to accumulation the excessive amount of dispersant molecules around of particles. The excess of dispersant would hinder movement of particles, leading to agglomeration of particles due to surface tension effect of dispersant [20]. On the other hand, high dispersant concentrations result in a significant shift in pH value that increases the ionic strength of the suspension and the slip viscosity [21]. Moreover, the increase of the dispersant concentration leads to compression of the diffuse layer and to lowering of the ζ-potential value. The suspensions obtained using Darvan 821 A and Darvan C-N are less stable: the sediment mass at 0.6–1.5 mass.% dispersant concentrations exceeds 0.5 g (curves 2 and 3, respectively).

Fig. 5 presents the microphotograph of the particles in the stable suspension obtained under the optimum conditions: the Lu_2O_3 solid content is 5, 10 vol.%, the Dolapix CE 64 concentration is 1 wt.%, the suspension pH is 9–10, the duration of homogenization in a ball mill is 40 h, the mass ratio of the grinding bodies to that of the powder being ∼4:1. The suspension is most stable when dispersant is fully dissociated. Dispersants on the base of polymethacrylic and polyacrylic acid (such as Dolapix CE 64, Darvan 821 A, Darvan C-N) completely dissociate at pH about 9 [22,23], thus providing a basis for obtaining of stable suspension. Since the specific

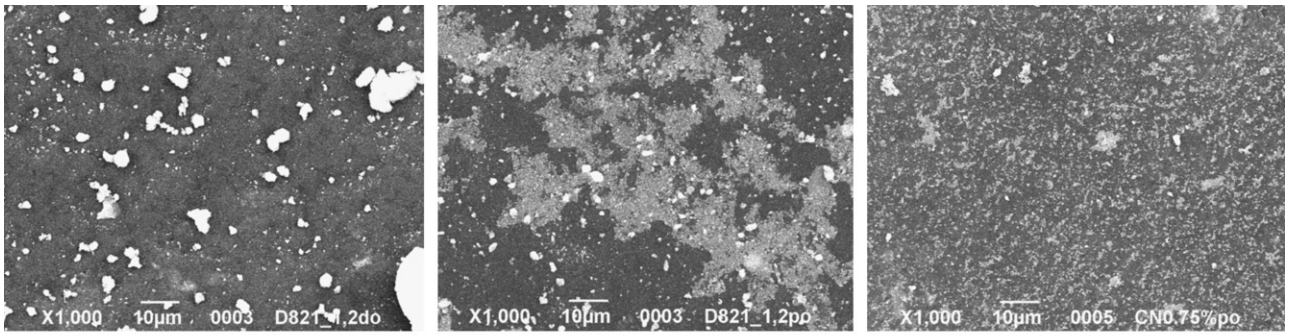

Fig. 3. The particle aggregation state after suspension drying: 1, before homogenization; 2, after 20-h homogenization; and 3, after 40-h homogenization.

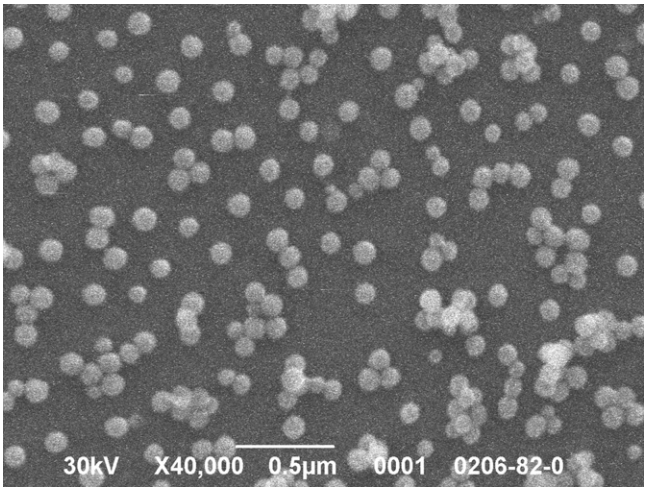

Fig. 5. The particle aggregation state after Lu_2O_3 stable suspension drying.

surface area of the powder is 20 m^2/g, then 10 mg/g of dispersant concentration is equivalent to 0.5 mg/m^2. It should be noted, that such a dispersant concentration is typical for other stable suspensions [8]. Thereat, the suspension viscosity is minimal and makes 1.7–2.0 cP which is close to the viscosity of pure water (1.005 cP). Such viscosity values satisfy the requirements for the slurry and testify to the fact that the suspension particles are not linked and move independently. This is confirmed by Fig. 5.

3.4. Effects of dispersant type and concentration on the density of green bodies

The slip casting method was used to prepare green bodies from stable Lu_2O_3:Eu^{3+} slurry. Then the obtained compacts were dried in air at room temperature in order to remove residual moisture. Fig. 6 shows the dependence of the relative density of the compacts on the dispersant type and its content in the slurry at a constant pressure of 138 MPa. The lower the molecular mass of the used dispersant is the more dense compacts are formed by pressure filtration. Dispersant with a higher molecular mass has a longer hydrocarbon chain which may be adsorbed on several particles, but not on one, thus giving rise to flocculation. The latter may cause inhomogeneity of the packing and insufficient density of the compact. The compact obtained from the slurry containing 1 mass.% of Dolapix CE 64 has the highest density of 51% of the theoretical value. This value is the same (within the density measurements error) as the calculated density for the primitive cubic packing of the spherical particles (52%). Further rise of the dispersant concentration leads to their excess accumulation in the interparticle space, that preventing the particles molding.

The relative density of green bodies obtained from the dispersed slurry (1 mass.% of Dolapix CE 64) and flocculated slurry (containing excessive dispersant in concentration of 1.5 mass.%) are presented in Fig. 7 (samples 1 and 2, respectively). In the flocculated slurry the dispersant excess results in the formation of aggregates where the particles are separated from each other by water layers. The density of green bodies obtained from such suspensions essentially depends on the pressure applied and increases within the 69–140 MPa pressure range. Such a behavior can be explained by displacement and rearrangement of the particles in the slurry: destruction of the agglomerates, water interlayers removal and breakdown of the bridges between the particles. The relative density of the dispersed slurry is not sensitive to the pressure applied due to the fact that the particles are not agglomerated and move independently, as in Newtonian liquid [24]. The density of compacts obtained from dispersed slurry is much higher compared to flocculated one irrespective of the pressure, and reaches the value of 51%. The specific hydraulic resistance of the sediment which is main filtrating layer considerably increases with increase the compact thickness and density during dehydration process. However, dense crackles Lu_2O_3:Eu^{3+} compacts can be obtained at relatively low pressures of 70 MPa when the dispersed slurry is used.

The presence of organic additives in the compact during vacuum sintering may lead to the formation of defects due to the intense degassing upon water evaporation or the organic compounds decomposition. Therefore, we conducted a preliminary annealing of compacts in an oxidizing environment at the T = 1200 °C for 2 h to remove organic additives from the

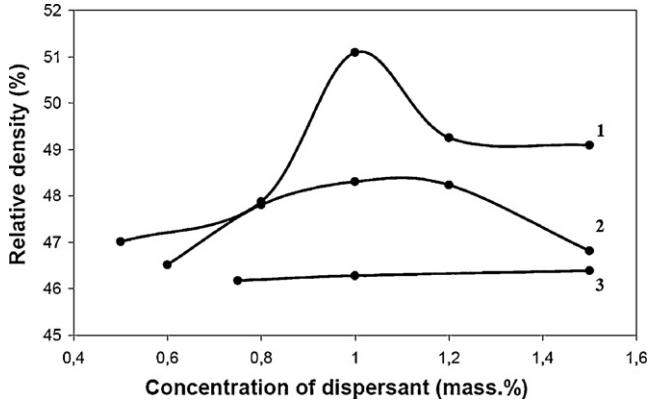

Fig. 6. Dependence of the relative density of Lu_2O_3:Eu^{3+} compacts dried at room temperature on the type and concentration of the dispersants: 1, Dolapix CE 64; 2, Darvan 821 A; 3, Darvan C-N.

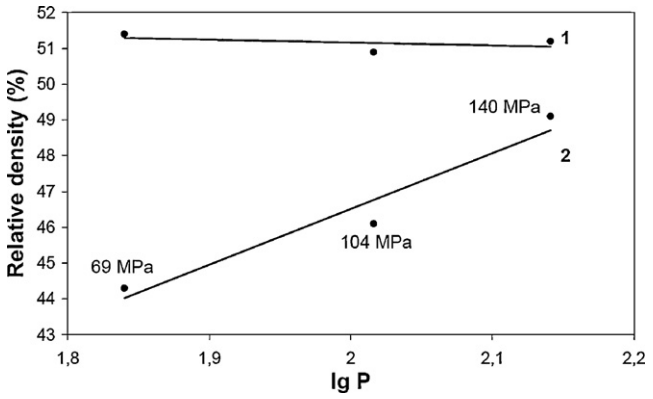

Fig. 7. Relative density of compacts as a function of the logarithm of the applied pressure: 1, dispersed slurry (1 mass.% Dolapix CE 64); 2, flocculated slurry (1.5 mass.% Dolapix CE 64).

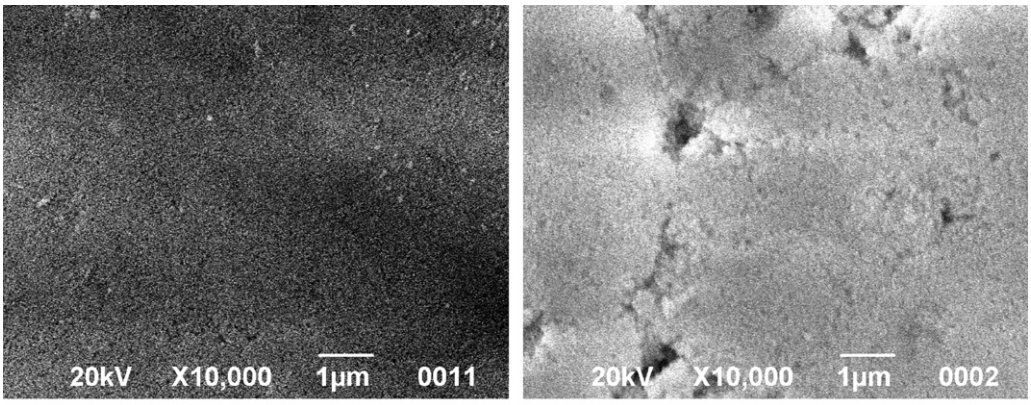

Fig. 8. Microstructure of Lu_2O_3:Eu^{3+} compacts obtained at the pressure of 150 MPa by: 1, slip casting; 2, uniaxial pressing.

compact. Chemical analysis of the compacts after annealing confirmed the absence of traces of organic additives.

After the annealing of the organic additive, the resulting density of the compact Lu_2O_3:Eu^{3+} containing 1 mass.% of Dolapix CE 64 is equal to 52% of the theoretical value. This value is considerably higher compared to 45% for Lu_2O_3:Eu^{3+} nanopowders molded by cold isostatic pressing under 200 MPa pressure [25]. Moreover, compacts obtained by the pressure slip casting are characterized by uniform particle packing and density distribution, in contrast to the ones obtained by the uniaxial pressing method. This is clearly seen from Fig. 8. Similar peculiarities were observed during molding of Y_2O_3 nanopowders [4]. Non-uniform density distribution in the dry pressed compacts (Fig. 8, sample 2) may lead to locally different shrinkage on subsequent sintering which gives rise to deformation and cracking. We have not measured the pore size distribution in the Lu_2O_3 compacts obtained, but according to data for Al_2O_3 [26], only slip casting method provides the homogenous close packed microstructure with minimal pore size.

The molded compacts obtained by the pressure slip casting were used to produce Lu_2O_3:Eu^{3+} translucent ceramics. The polished 0.5 mm thick sample of transparent Lu_2O_3:Eu^{3+} ceramics prepared by vacuum sintering at 1800 °C for 5 h is presented in Fig. 9. The green body was produced using stable slurry containing 1 mass.% of Dolapix CE 64. Increase of the Lu_2O_3:Eu^{3+} solid loading in the slurry to obtain the compacts with higher initial density of 55–60% is an object of our further study.

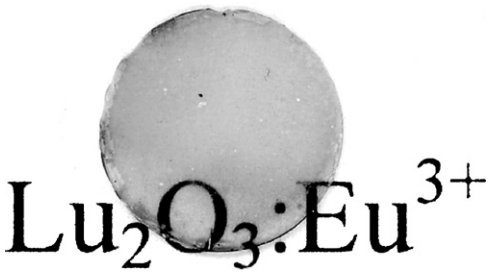

Fig. 9. Appearance of mirror-polished Lu_2O_3:Eu^{3+} transparent ceramic specimen (the thickness is 0.5 mm).

4. Conclusions

The influence of the commercial available dispersants Dolapix CE 64, Darvan 821 A and Darvan C-N on the stability of Lu_2O_3:Eu^{3+} colloid suspensions has been studied. The dependence of the stabilizing effect of the dispersants on their molecular weight and concentration has been established. The performed measurements of the sedimentation properties and the density of the compacts obtained by the slip casting method show that among the studied dispersants Dolapix CE 64 is most efficient. The optimal conditions to obtain stable suspension containing 5, 10 vol.% of Lu_2O_3:Eu^{3+} have been determined: the concentration of Dolapix CE 64 is 1 mass.%, the suspension pH is 9–10, the duration of homogenization in a ball mill is 40 h, the mass ratio of the grinding balls to that of the powder being ∼4:1. The pressure slip casting was used to prepare compacts with a maximum density of 52% of the theoretical value and uniform density distribution. The compacts obtained were used to produce Lu_2O_3:Eu^{3+} translucent ceramics by the vacuum sintering method.

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