

Optimization of rheological properties of Y_2O_3 slurries for obtaining IR-transparent ceramics

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The effect of dispersant concentration on the sedimentation stability of aqueous suspensions of Y_2O_3 nanopowders, as well as the influence of the slurry composition, such as the dispersant content and solid loading, on their rheological properties were investigated. It was found that 30 wt.% Y_2O_3 and 1.5 wt.% Dolapix CE64 water suspension with the highest solid loading has rheological properties close to Newtonian fluids and low viscosity. Increasing the dispersant or solid content leads to increase in slurry viscosity and loss of Newtonian behavior. Hemispherical samples of infrared transparent Y_2O_3 ceramics were obtained by the slip casting method followed by vacuum sintering at 1750°C. The obtained ceramics are characterized by a relative density of 99±1 %, the average grain size of 10–15 μm , and in-line transmittance of 30 % and 63 % at the wavelengths of 800 and 2000 nm, respectively.

Keywords: slip casting, dispersant, yttrium oxide, transparent ceramics.

Оптимізація реологічних властивостей шлікерів Y_2O_3 для отримання ІЧ-прозорої кераміки. Д.Г.Черноморець, О.С.Крижановська, Н.А.Сафронова, А.Е.Балабанов, А.Г.Дороженко, І.О.Ворона, С.В.Пархоменко, О.В.Толмачов, Р.П.Явецький

Досліджено вплив концентрації дисперсанта седиментаційну стабільність водних суспензій нанопорошків Y_2O_3 , а також вплив складу суспензії, такого як вміст дисперсанта та твердої речовини, на їх реологічні властивості. Встановлено, що водна суспензія 30 мас.% Y_2O_3 і 1,5 мас.% Dolapix CE64 з найбільшим вмістом твердої речовини має реологічні властивості, близькі до ньютоновських рідин, і низьку в'язкість. Збільшення вмісту дисперсанта або твердої речовини призводить до збільшення в'язкості суспензії та втрати властивостей ньютонівських рідин. Напівсферичні зразки інфрачервоної прозорої кераміки Y_2O_3 отримували методом шлікерного ліття з подальшим вакуумним спіканням при 1750°C. Отримана кераміка характеризується відносною щільністю 99±1 %, середнім розміром зерна 10–15 мкм і лінійним оптичним пропусканням 30 % і 63 % на довжинах хвиль 800 і 2000 нм відповідно.

1. Introduction

Nowadays yttrium oxide ceramics are intensively studied and founds practical application as transparent windows, laser media, refractory components and semiconductor devices. Y_2O_3 ceramics are characterized by lower emissivity and emittance in the infrared (IR) range at high temperatures in comparison

with spinel, sapphire and aluminum oxynitride (AlON). Thus, this material is a promising one for utilization as IR-transparent windows which operate at elevated temperatures under extreme operating conditions [1–4].

Obtaining of highly-transparent ceramics requires a complete elimination of scattering centers, such as impurity phases and residual pores after consolidation step, for

example, by vacuum sintering [5]. The compaction process is one of the most important stages in the transparent ceramics production, which allows to obtain high density green bodies with a homogeneous microstructure. The homogeneous microstructure of the green bodies with uniformly distributed pore channels precisely enables high sinterability of ceramics. Currently, the most common forming methods of initial powders are the cold isostatic pressing (CIP), or a combination of uniaxial pressing with CIP [6–7]. The dry pressing methods meets some difficulties in obtaining homogeneous compacts from nanopowders [8]. Therefore, wet forming methods, such as slip casting, were proposed as an alternative to dry pressing methods in order to achieve excellent homogeneity via self-organization of powder particles in suspensions. The utilization of liquid phase during slip casting reduces the interparticle friction forces and promotes the formation of capillary forces that provides dense packing of nanoparticles [9–10]. Moreover, slip casting allows to form homogeneous highly-dense green bodies of complex geometry, which expands the possible application areas of ceramics in contrast to the dry pressing methods.

Controlling the suspension parameters during slip casting is an important goal of ceramic processing. Slurries for slip casting should meet the following requirements: high solid loading, low viscosity value, and the Newtonian fluid behavior. Fulfilment of these conditions provides highly-dense compacts possessing an uniform distribution of nanoparticles [11]. Preparation of stable highly-loaded slurries with a low viscosity using nanopowders is a complex technological task since the high surface energy of particles contributes to their agglomeration and even a loss of the slurry stability. In order to effectively stabilize nanopowders in the suspension the dispersants are used. Dispersants are surfactants or polyelectrolytes that increase the suspension stability while adsorbed on the particles surface [12]. In this work, the anionic polyelectrolyte Dolapix CE64 was used as a dispersant, which provides effective stabilization of the slurries based on the oxide nanopowders [2, 13–15]. This work aimed to study the effect of dispersant and solid loading on the stability and rheological properties of Y_2O_3 suspensions and to obtain hemispherical samples of IR-transparent Y_2O_3 ceramics.

2. Experimental

Y_2O_3 powders (Alfa Aesar, nanopowder, 99.995 %) were used as the starting materials. ZrO_2 (99 %, US Research Nanomaterials), Yb_2O_3 (99.99 %, Alfa-Aesar), and La_2O_3 (99.99 %, Alfa-Aesar) were used as sintering additives. The initial powders were mixed in $(\text{Y}_{1.86}\text{Yb}_{0.10}\text{La}_{0.01}\text{Zr}_{0.03}\text{O}_3)$ stoichiometric ratio (further denoted as Y_2O_3) and homogenized with isopropanol in a Fritsch Pulverisette 5/4 planetary ball mill for 24 hours. The rotation speed was 140 rpm, ZrO_2 jars and balls were used. After homogenization the slurry was dried followed by subsequent annealing at 600°C for 4 hours. Commercial water-soluble polyelectrolyte Dolapix CE64 (Zschimmer & Schwartz GmbH, Germany) (ammonium polymethacrylate with a molecular weight of 350 g/mol) was added as a dispersant in various concentrations to ensure slips stability. The deionized water was used as a solvent, and the pH of the suspensions was maintained in the range of 9–10 by adding ammonium hydroxide solution (NH_4OH). To study the effect of solid loading on the rheological characteristics of slips the solid loading was varied in the range of 30–35 wt.% at the constant dispersant concentration of 1.5 wt.%.

To study the effect of the dispersant content on the stability and rheological characteristics of slurries, compositions containing 1.5, 1.75 and 2.0 wt.% of Dolapix CE64 at solid loading 30 wt.% were prepared. These slips were prepared by homogenization of the nanopowders, deionized water and dispersant in a planetary ball mill for 22 hours. The dispersant mass was calculated based on the powder mass. Homogenization was performed at a ball/solids mass ratio of 3/1. To remove air bubbles remaining after the homogenization process, the suspension was evacuated for 10–20 minutes.

The suspension stability was determined by the sedimentation column height in the suspensions after 24 hours. According to the obtained data, the optimal dispersant content was determined. The suspensions viscosity was measured using a rotary viscometer RM 100 Plus Lamy Rheology with a measuring system MS DIN 19 Lamy Rheology.

Samples compaction was performed by casting the above-described slips in hemispherical gypsum molds with a diameter of 50 mm and subsequent drying. After removing the compacts from the mold, the

green bodies were annealed in air at 800°C for 4 hours in order to remove organic impurities. Consolidation of compacts was carried out by vacuum sintering at 1750°C for 10 hours.

Electron microscopic studies were performed by the scanning electron microscopy (JSM-6390 LV, JEOL, Japan). Optical transmission spectra of polished samples with a thickness of 1.8 mm were measured using a Lambda-35 spectrophotometer (Perkin-Elmer, USA) in the wavelength range 200–1100 nm. Infrared spectra were recorded using a Spectrum One (Perkin-Elmer, USA) IR-Fourier spectrophotometer in the 400–4000 cm⁻¹ range. Microstructural studies of ceramics were performed using a Zeiss PrimoTech optical microscope with a magnification of up to $\times 1600$ in the reflected light (surface and grain structure) and transmitted light (volume structure and residual pores) modes. Vickers microhardness measurements of ceramics were made using a PMT-3 hardness tester. The load $P = 1$ N was used for indentation time of 10 s. The relative density of green bodies and ceramics was estimated by the hydrostatic weighing method.

3. Results and discussion

3.1 Influence of dispersant content on stability of Y₂O₃ slurries

Nanopowders' particles in suspension tend to aggregate due to high surface energy. It has a negative effect on the stability of the slurries, as the agglomerates settle faster and create inhomogeneities in the compact. Dispersants allow to increase the slurries' stability by preventing the agglomeration of particles and ensuring their independent movement from each other.

Dolapix CE64, a commercial salt of polymethacrylic acid with a molecular weight of 350 g/mol, was used to obtain stable slurries. Dolapix CE64 is an anionic polyelectrolyte which contain ionogenic carboxyammonium groups $-\text{COOH}_4^-$. The hydrolysis of these groups in an alkaline environment leads to the formation of negatively charged carboxylate polyions (COO^-) and ammonium ions [14, 16]. It is known that Dolapix CE64 starts to dissociate at pH about 3.5 and completely dissociates at pH above 8.5 as an anionic dispersant [2, 13]. The addition of dispersants significantly affects the zeta potential of suspensions; the isoelectric point of yttrium oxide is shifted from 4 (for suspension without dispersant) to about 8.7 (with dispersant). This is due to the adsorption of the dispersant on the

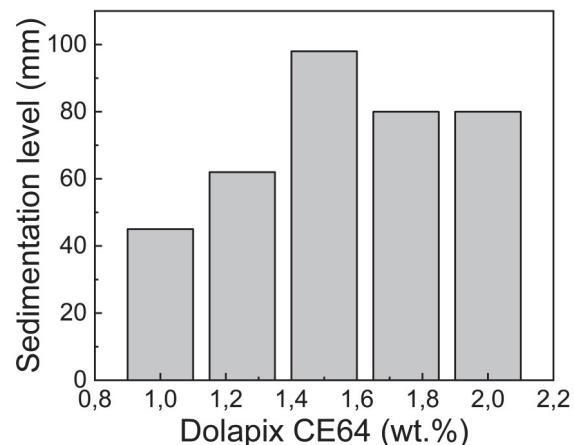


Fig. 1. Sedimentation column height of 10 wt.% Y₂O₃ slurry containing different concentration of Dolapix CE64

surface of the yttrium oxide particles. Moreover, the absolute value of the zeta potential of the suspension with the dispersant, and hence the maximum stabilization effect, corresponds to a pH value of about 10 [9]. Therefore, the pH of the suspension was maintained at the required level by adding NH₄OH solution.

It is necessary to consider the reaction between water and Y₂O₃, as this may affect the properties of the suspension during the slip casting process. It is known that the adsorption of water molecules on the surface of yttrium oxide particles leads to the formation of Y₂O₃(H₂O) or YOOH-like particles, resulting in formation of Y(OH)₃ [17]. Stabilization of suspension occurs because the polyions of the dispersant are drawn into the adsorption layer of the particle due to strong electrostatic interactions and the high adsorption capacity associated with polarization of such ions [2, 13].

The dispersant's concentration effect on the stability of suspensions was studied by sedimentation test. Suspensions with a constant powder content (10 wt.%) and a dispersant concentration in the range of 1.0–2.0 wt.% were dispersed by sonication for 15 minutes, the suspension level was measured after 24 hours. Fig. 1 shows the dependence of the height of the sedimentation column on the dispersant content. It was found that the suspension with 1.5 wt.% of dispersant is the most stable (column height is 98 mm). Increasing the content of dispersant to 1.75–2.0 wt.% leads to a decrease in the stability of suspensions (column height is 80 mm), since an excess of dispersant promotes aggregation of particles and faster sedimentation. Insufficient amount

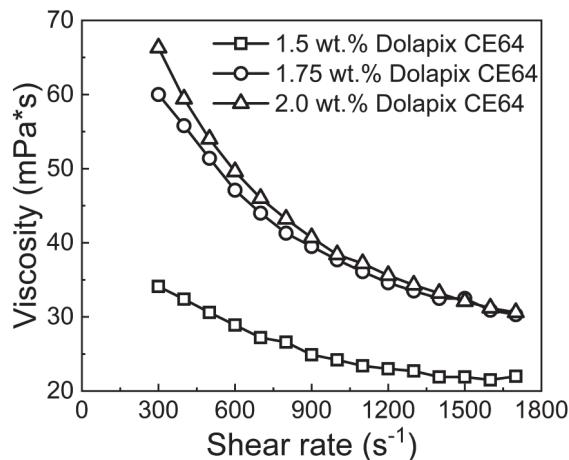


Fig. 2. The dependence of viscosity on the shear rate of 30 wt.% Y_2O_3 suspensions containing 1.5, 1.75, and 2.0 wt.% of Dolapix CE64

of dispersant adsorbed on the particles' surface will lead to their agglomeration and loss of stability; thus, reducing the concentration of Dolapix CE64 down to 1.25 wt.% seems to be inappropriate.

3.2 Effect of dispersant concentration on rheological characteristics of Y_2O_3 slips

Based on sedimentation tests, we studied the rheological characteristics of Y_2O_3 slurries with Dolapix CE64 content in the range of 1.5–2.0 wt.% at the 30 wt.% of solid loading (Fig. 2). Fig. 2 shows that the lowest viscosity values were obtained for a suspension with a dispersant content of 1.5 wt.%. It can be seen that the viscosity of this suspension varies slightly with the shear rate and reaches 20–30 MPa·s for a shear rate in the 300–1700 s⁻¹ range. The obtained data indicate that the water suspension behaved near Newtonian, and the dispersant has a double stabilizing effect: electrostatic and spatial repulsion of particles due to adsorption on their surface. Adsorption of the dispersant increases the charge density of the particles, which leads to the thickening of the electric double layer and increases the electrostatic repulsive force between the particles in the suspension.

Dispersant adsorbed on the surface creates a strong spatial repulsion, which in turn could overcome the Van der Waals intermolecular attraction forces between particles (steric stabilization) [19–21]. Most likely, for 1.5 wt.% of Dolapix CE64 the surface of yttria particles is tightly covered by adsorbed anions of dispersants. This ensures an optimal electrosteric repulsion of

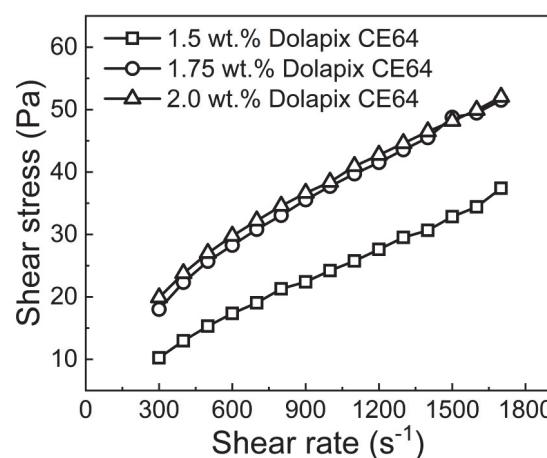


Fig. 3. Dependence of shear stress on shear rate of 30 wt.% Y_2O_3 suspensions containing 1.5, 1.75, and 2.0 wt.% of Dolapix CE64

particles owing to free stretching of polyelectrolyte chains. Thus, the most stable suspension with a minimal viscosity is formed [5, 9]. Further increase of the dispersant concentration to 1.75 and 2.0 wt.% results in almost 2-fold increase of suspension viscosity (Fig. 2) due to oversaturation of Y_2O_3 surface with dispersant. Excess of Dolapix CE64 molecules in the suspension's environment leads to their interaction having viscosity increase as a result [22]. Recently it was shown [14] that slurry viscosity variation with increasing dispersant content is explained by a change in pH value, as well as increase the ionic strength of the slurry. Reducing the concentration of Dolapix CE64 below 1.5 wt.% doesn't make a sense because adsorption of fewer dispersants amount on the particle surface leads to the formation of thinner double electric layer. This may cause the particles to be electrostatically attracted to each other, reducing the homogeneity of the suspension and viscosity increase.

The rheological characteristics of slurries were studied by analyzing the flow curves — the dependence of shear stress on shear rate for 30 wt.% Y_2O_3 suspensions with a dispersant content in the range of 1.5–2.0 wt.% (Fig. 3). As one can see the flow curve for the suspension containing 1.5 wt.% of Dolapix CE64 is linear and passes close to zero, so it is close to the Newtonian fluid. The nature of the dependence indicates that suspension is homogeneous and well-dispersed. The flow curves for suspensions containing 1.75 and 2.0 wt.% Dolapix CE64 are similar and correspond to flocculated pseudoplastic structurally-vis-

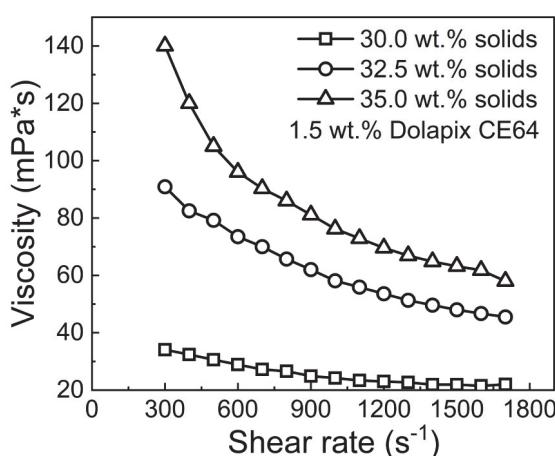


Fig. 4. Dependence of viscosity on shear rate of Y₂O₃ suspensions containing different solid loading (1.5 wt.% Dolapix CE64)

cous suspension. In 2.0 wt.% Dolapix CE64 suspensions strongly anisotropic polymer chains of the dispersant begin to unravel and align in the direction of shear at a high shear rate (Fig. 3). This leads to lower interaction of molecules and particles and provides more free space; the viscosity of suspension is reduced providing a shear behavior. Therefore, the shear diluted flow leads to the destruction of the flocculated structure of the particles with the dispersant and the release of trapped liquid [23]. Thus, the analysis of the rheological properties of 30 wt.% yttrium oxide suspension revealed that the optimal concentration of Dolapix CE64 dispersant is 1.5 wt.%; the suspension behaves similar to the Newtonian fluid and the dispersant exhibits effective stabilization.

3.3 Influence of solid loading on rheological characteristics of Y₂O₃ slips

Suspensions with a high solid loading are required to produce a dense green body, since during the suction of liquid by gypsum compacts are formed quite quickly. Thus, an insufficient solid loading or excess of liquid in the suspension can lead to formation of pores. As a result, residual pores will survive in the sintered ceramics after vacuum sintering, affecting their optical properties [11, 24]. As the nanopowders' content of the slurries increases, their viscosity will increase either. Therefore, the preparation of a highly-loaded suspension requires a compromise between increasing the solid content and obtaining a low-viscosity slurry for casting process.

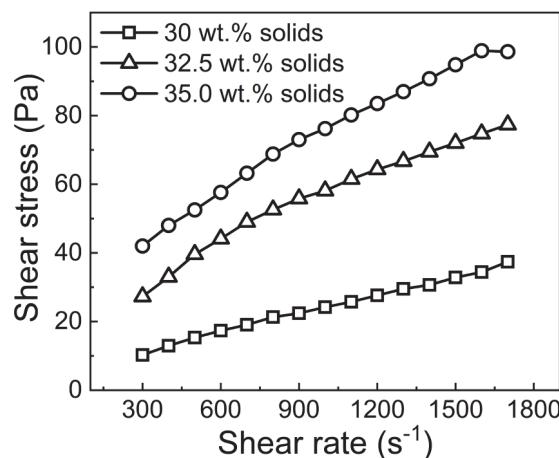


Fig. 5. Shear stress vs. shear rate of Y₂O₃ suspensions with different solid content of 30; 32.5, and 35 wt.% (1.5 wt.% Dolapix CE64)

Effect of solid loading on the rheological characteristics of Y₂O₃ slurries was investigated in the 30–35 wt.% concentration range of solid at constant dispersant concentration (1.5 wt.%). Let's consider the viscosity of the suspension obtained at different shear rates (Fig. 4). The viscosity of the 30 wt.% suspension is 25–30 MPa·s and does not depend on the shear rate in the measurement range studied (300–1700 s⁻¹). This indicates its similarity to Newtonian fluids. By increasing the nanopowder content up to 32.5–35 wt.% the viscosity of the suspensions increases (Fig. 4), and the slurry shows pseudoplastic properties due to the interaction of particles with each other.

Let's analyze the viscosity of Y₂O₃ suspensions containing different solid loading of 30, 32.5, and 35 wt.% and the constant Dolapix CE64 concentration of 1.5 wt.% (Fig. 5). As it was noted earlier, the suspension with solid loading of 30 wt.% has rheological properties the closest to Newtonian fluid. Slurries with higher solid content (32.5 and 35.0 wt.%) exhibit the properties of pseudoplastic fluid due to forced convergence between the nanoparticles. As a result, their polymeric shells begin to interact, leading the system to loss the Newtonian fluid properties [25]. Thus, 30 wt.% Y₂O₃ and 1.5 wt.% of Dolapix CE64 is the most highly-loaded slurry exhibiting rheological properties similar to Newtonian fluid and having a low viscosity.

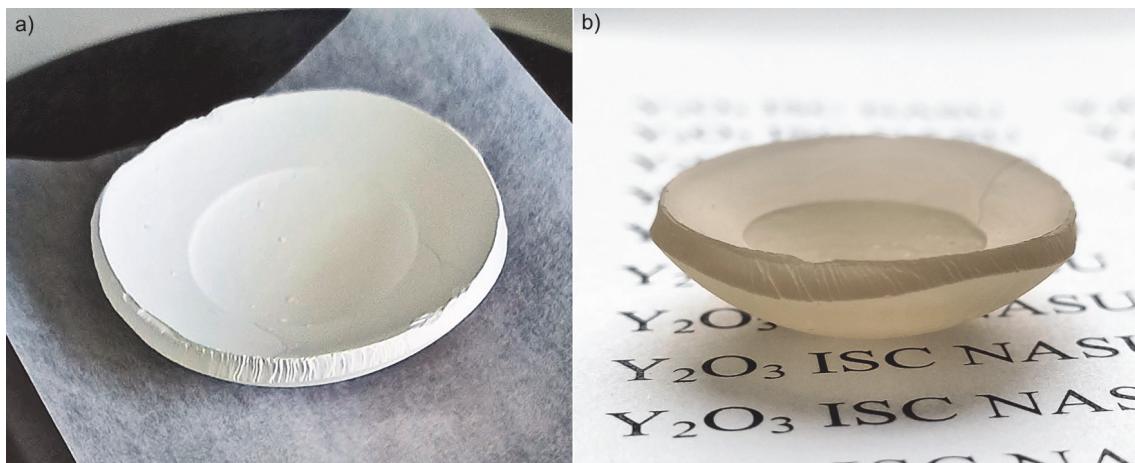


Fig. 6. Appearance of yttria green body (a) and transparent ceramics (b) obtained by slip casting followed by vacuum sintering at 1750°C for 10 hours

3.4 Processing and characterization of Y_2O_3 ceramics

Y_2O_3 ceramics were obtained by slip casting of slurries of optimized composition 30 wt.% Y_2O_3 /1.5 wt.% Dolapix CE64 into gypsum molds and subsequent vacuum sintering of compacts at 1750°C for 10 hours. Fig. 6 shows the appearance of hemispherical samples of Y_2O_3 green bodies and ceramics before and after vacuum sintering. The initial compacts had a relative density of 45.2 % from the theoretical value. The sintered ceramics are characterized by yellowish color connected with formation of color centers (mainly oxygen vacancies) after vacuum sintering. The coloration disappears after subsequent air annealing of ceramics in an air atmosphere.

Figure 7 shows the microstructure of yttrium oxide ceramic samples obtained using a slip of optimized composition (30 wt.% Y_2O_3 /1.5 wt.% Dolapix CE64). We see that Y_2O_3 ceramics are characterized by a quite homogeneous morphology, the average grain size is 10–15 μm . Single residual pores are observed in the sample, located both at the grain boundaries and in the volume of the grains, which act as main light scattering centers.

The relative density of Y_2O_3 ceramics is 99 ± 1 % from the theoretical value (5.23 g/cm^3). Achieving near the theoretical density after vacuum sintering may indicate a dense packing of starting nanopowders during slip casting process, as well as the high sinterability of the obtained green bodies. The microhardness of the obtained transparent yttria ceramics is 9.3 GPa which is comparable to that of ceramics ob-

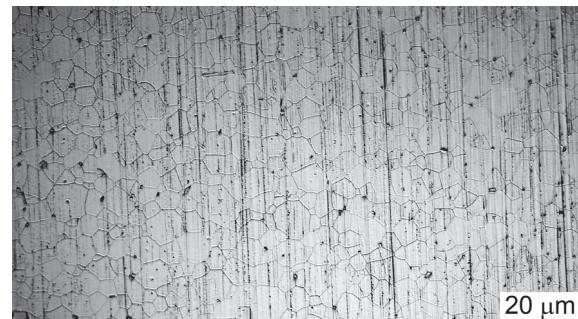


Fig. 7. Microstructure of Y_2O_3 ceramics obtained by slip casting and subsequent vacuum sintering at 1750°C for 10 hours

tained by cold isostatic pressing and vacuum sintering.

Fig. 8 shows optical properties of Y_2O_3 ceramics in the visible and infrared wavelength ranges. The in-line optical transmittance of the samples is ~30 % at the wavelength of 800 nm, and then increases significantly and reaches ~63 % at the wavelength of 2000 nm, which is 76 % of the theoretical value. The sharp decline of the transmittance in the visible wavelength range may be due to the presence of submicron pores in the samples, as evidenced by modeling the light scattering by spherical pores in transparent oxide ceramics [26]. Absorption peaks in the $\lambda = 800\text{--}1100 \text{ nm}$ range correspond to ${}^2\text{F}_{7/2}\text{--}{}^2\text{F}_{5/2}$ transitions in trivalent ytterbium ions [27–28]. The optical properties of Y_2O_3 ceramics prepared from the optimized suspensions are quite high, however, still below the values reported [28]. However, for practical application of ceramics as IR-windows of technological apparatus and vehicles the in-line optical transmittance reached seems to be

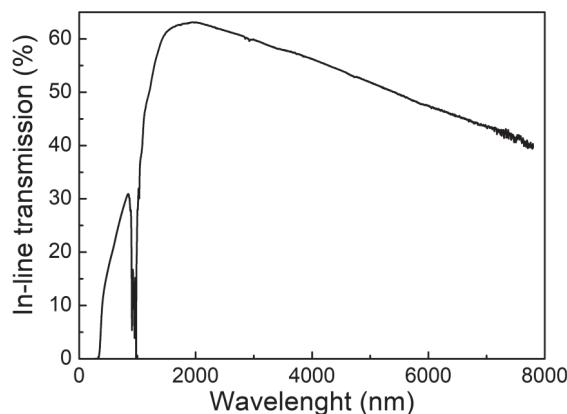


Fig. 8. In-line optical transmittance spectrum of $\text{Yb}^{3+}\text{:Y}_2\text{O}_3$ ceramics obtained by slip casting from an optimized slurry (30 wt.% Y_2O_3 /1.5% wt.% Dolapix CE64). The sample thickness is 1.8 mm

satisfactory. It should be noted that optical transmittance of yttria ceramics prepared from non-optimized slurries are much lower. The particle agglomeration in unstabilized slurries is likely to contribute to the packing defects formation and voids in green bodies, leading to incomplete densification and preservation of high residual porosity in sintered ceramics [5, 27].

4. Conclusions

Sedimentation stability of aqueous suspensions of yttrium oxide nanopowders depending on the content of Dolapix CE64 dispersant was investigated. It was found that slip containing 1.5 wt.% of dispersant is the most stable. At this concentration, the polyions formed by dissociation of the Dolapix CE64 fully cover the surface of the nanoparticles and can stretch freely. This leads to optimal electrosteric repulsion of the particles and, therefore, to the stability of powder suspensions of yttrium oxide. Increasing the concentration of the dispersant to 1.75 and 2 wt.% leads to flocculation, i.e., to the loss of stability of the suspension due to the formation of agglomerates as a result of the interaction of the polymer chains of the dispersant.

The effect of the slip compositions (solid loading and dispersant concentration) on their rheological properties was determined. The analysis of slurry flow curves shows that 30 wt.% Y_2O_3 and 1.5 wt.% of Dolapix CE64 is the most highly-loaded slurry exhibiting rheological properties similar to Newtonian fluid and having a low viscosity. Slurries with higher dispersant concentration or higher solid loading have increased

viscosity and exhibit pseudoplastic properties due to forced convergence between the nanoparticles.

IR-transparent yttrium oxide ceramics of hemispherical shape were obtained by slip casting followed by vacuum sintering at 1750°C. The samples are characterized by the relative density of 99±1 %, homogeneous microstructure and average grain size of 10–15 µm. The in-line optical transmittance of ceramic samples obtained from the slurry of optimized composition (30 wt.% Y_2O_3 /1.5 wt.% Dolapix CE64) is 30 and 63 % at the wavelengths of 800 nm and 2000 nm, respectively.

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