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LETTERS TO THE EDITOR

Features of the Synthesis of Nanoparticles of Yttrium Oxide Y_2O_3:Nd

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Active medium of solid-state lasers, laser ceramics based on yttrium oxide, has a combination of properties providing its high competitive ability relative to both laser glasses and laser crystals. Properties of source materials, like chemical and phase purity, particle size, particle size distribution, homogeneity of particle shapes, and absence of rigid agglomeration, belong to critical factors of manufacturing transparent ceramics [1].

Investigation of the synthesis of yttrium oxide (Y_2O_3 : Nd) by the Pechini method has allowed us to modify the procedure with the purpose of improving properties of resulting powders, precursors of optical ceramics.

In this work Y_2O_3 : Nd powders are considered from the viewpoint of morphology and agglomeration.

In the late 1960th it was suggested to use the method including a preliminary stage of the reaction between initial components in solution resulting in the gel formation and in the decomposition of formed metal-polymer compositions up to oxides for the synthesis of inorganic compounds [2]. The gel is formed as a result of the esterification reaction between a polyatomic acid, which plays the role of a ligand for metal ions, and a polyatomic alcohol. Citric and ethylenediaminetetraacetic acids and polyethylene glycol are the most often used acid and alcohol, respectively. As a result of the thermal processing of the polymeric gel a porous mass consisting of strongly agglomerated nanoparticles of synthesized oxides is formed. The Pechini method is not applicable, for example, to the preparation of nanoparticles, which then should be transferred into solution with adding a stabilizer. To obtain an optical laser ceramics, the powder should be necessarily ground in a ball mill [3]. Therefore the aim of this work was to modify the Pechini method in such a way as to obtain less agglomerated powders, saving advantages of the method.

We modified the Pechini method, using the principle operating in the method of the self-propagating high-temperature synthesis, which makes it possible to obtain low-agglomerated nanocrystal powders Y_2O_3. In this case the temperature in a sample reached 900–1000°C, however no qualitative caking occurred. It is due to the fact that the self-propagating high-temperature synthesis is accompanied with intensive gasification of reagents, which prevents from the subsequent more complete sintering [4].

The product of the Pechini synthesis was obtained by a thermal treatment of the polymeric gel, which represented a structured colloid system. Solid particles of the disperse phase are connected with each other in a loose space net containing a liquid disperse medium in the meshes. Contacts between particles are destroyed under the action of heat during the gel calcination. We suggest to include a process of intensive gas evolution in this closing stage, which would hinder the agglomeration and caking of particles similarly to the principle of the self-propagating high-temperature method of the synthesis. To do that, it is necessary to add a foaming agent to the system before the polymer formation, which further will fill the

structural gel net. The foaming agent should meet several demands: it should not react with main components of the system (in this case, yttrium and neodymium oxides) or to form materials reacting with them; not hinder the complete synthesis proceeding; should be easily removable from the product with a solvent, which does not contaminate the target product.

We have chosen a mixture of aluminum nitrite and potassium chloride as such a component for the Y_2O_3 synthesis. When a citric acid excess was present in a solution, the reaction providing the uniform foaming of a mass proceeded in the course of the gel thermal treatment.

According to a thermodynamic calculation, potassium chloride reacts with citric acid on heating [Eq. (1)].

$$H_3C_6H_5O_7 + KCl \rightarrow KH_2C_6H_5O_7 + HCl. \quad (1)$$

Under subsequent calcination the synthesized potassium citrate dihydrate burns down in oxygen atomsphere [Eq. (2)].

$$4KH_2C_6H_5O_7 + 7O_2 = 2K_2CO_3 + 14H_2O + 22CO\uparrow, \quad T > 150°C. \quad (2)$$

Potassium carbonate reacts with aluminum oxide to form aluminate, which is removed from the sample on washing with distilled water [Eq. (3)].

$$Al_2O_3 + K_2CO_3 = 2KAlO_2 + CO_2\ (600°C). \quad (3)$$

Calcination of the polymeric gel, which contained uniformly distributed metal ions and the foaming agent, was carried out at 1000°C within 2 h. According to the electron microscopy data, Y_2O_3 obtained in such a way consists of low-agglomerated particles of the size 100 nm. The X-ray analysis confirms the presence of the unique crystal phase Y_2O_3.

Theoretically, the procedure allows obtaining simple oxides applicable as active laser ceramics (lutetium, gadolinium, scandium, etc. oxides).

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