XRD studies of the precursor thermal decomposition in the production of nanopowders $LaLuO_3:Yb^{3+}$ with a perovskite type structure

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INTRODUCTION

Since the invention of the first laser with a polycrystalline active medium in 1964 [1], scientists have been constantly searching for ways to improve its performance, requiring the development and research of new materials with given properties. Such materials require the development and advancement of powder nanotechnologies for the creation of transparent functional ceramics. Nowadays, great practical interest has been shown in materials based on rare earth elements (REE) phase with perovskite type structure. The interest in optical ceramics as laser, scintillation media is due to the high optical transparency in a wide range of wavelengths, radiation resistance, high thermal conductivity, good thermomechanical properties, chemical and thermal stability. Ceramics have high manufacturability, wide variation in chemical composition and can be obtained in the form of composite elements with different structure [2, 3].

EXPERIMENTAL

To obtain nanopowders of complex oxide phases $LnLn'O_3$ (Ln, Ln' = REE) with the perovskite type structure, precursors were synthesized by heterogeneous precipitation from nitrate solutions of rare-earth elements in ammonia solution with urea percentage of 5 vol. % and at the solution temperature of 80°C. The process of structure formation evolution during thermal decomposition of the synthesized precursor in obtaining complex perovskite type oxide phases in La_2O_3-Lu_2O_3-Yb_2O_3 system was determined by X-ray diffraction in CuKα radiation using powder method on diffractometer DRON-3M.

Two main steps were found. The first one is related to the amorphous structure: the decomposition of the complex chemical compound formed after the precipitation and its internal rearrangement, and the second one to the formation and accumulation of the perovskite crystalline phase.

X-ray spectra of the samples obtained at temperatures up to 700°C at the first stage of the decomposition contain two broad diffuse peaks (halo) at 22-38° and 38-70°. In the interval of precursor's thermal decomposition temperatures from 120 to 700°C the processes of complex compound decomposition and internal rearrangement take place. An amorphous powder is formed with a gradual increase in the atomic ordering, evidenced by a decrease and a qualitative change in the background (Fig. 1). Possibly ordered domains typical for REE carbides, hydrides or carbohydrides are formed in the amorphous matrix.

With further increase in temperature the second main stage of decomposition begins. The process of formation and accumulation of the perovskite crystal phase is not uniform. At 750-780°C the size of crystallites of the perovskite type phase $LaLuO_3:Yb^{3+}$ rapidly increases, after which the growth slows down (Fig. 3).

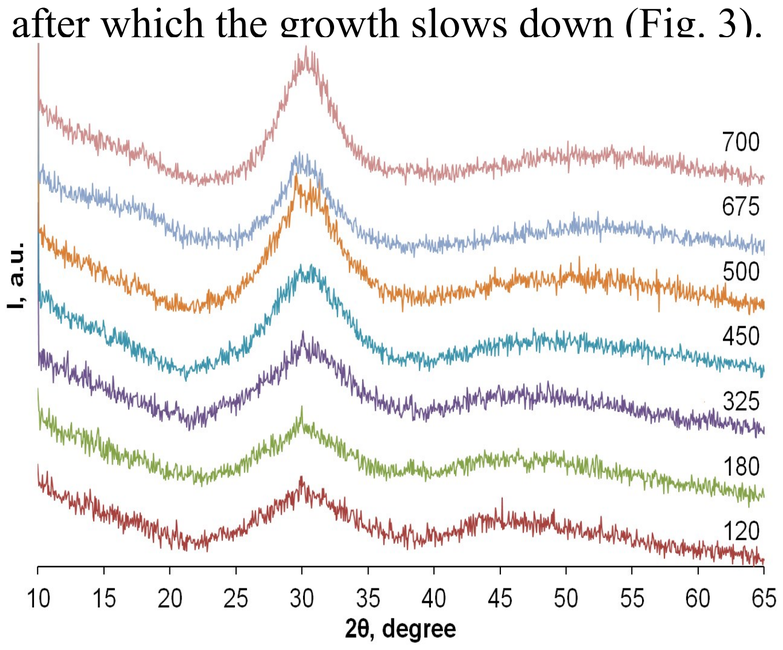

Figure 1. X-ray diagrams after precursor thermal decomposition up to 700°C

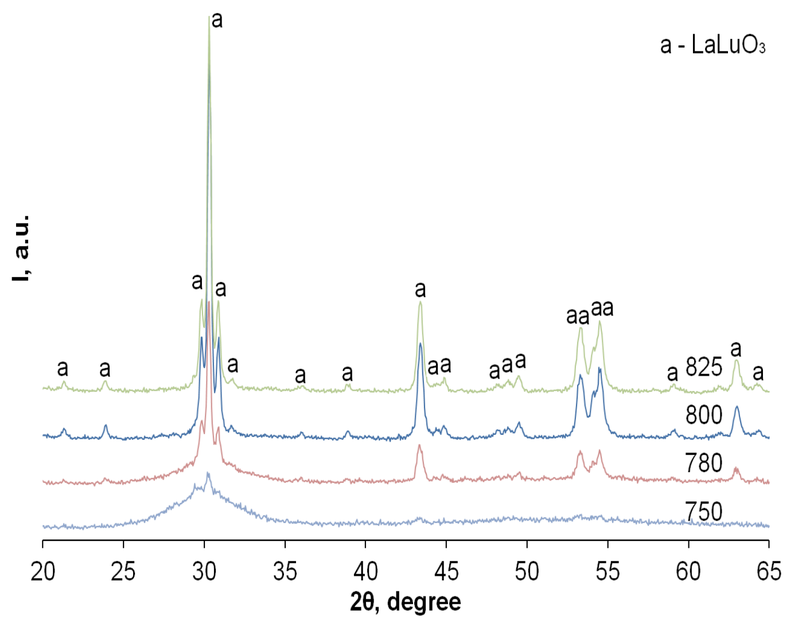

Figure 2. X-ray diagrams after precursor thermal decomposition 750-825°C

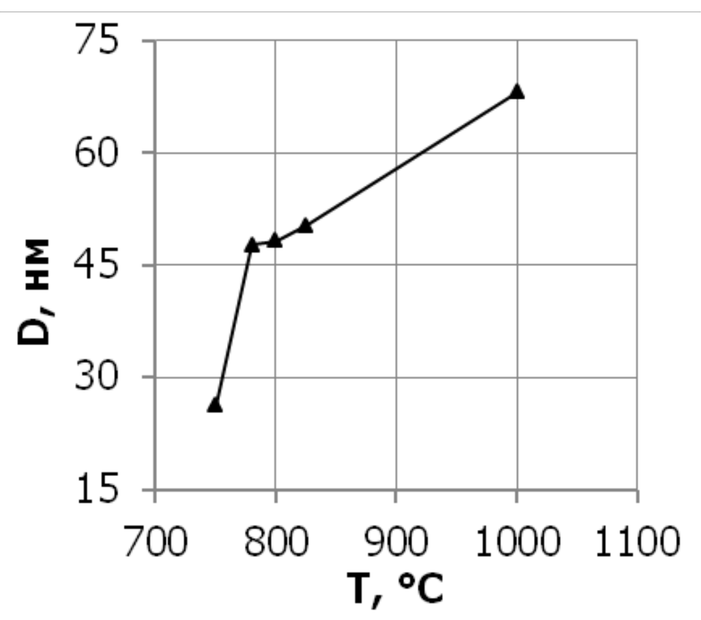

Figure 3. Plot of the precursor crystallite size temperature dependence

CONCLUSIONS

A study of the thermal decomposition products structure of the synthesized precursor under nonisothermal conditions of linear heating at a rate of 5 deg/min in obtaining $LnLn'O_3$ (Ln, Ln' = REE) nanopowder of complex oxide phases with a perovskite type structure showed that the synthesized precursor has the following main stages: decomposition of the complex chemical compound formed after deposition, intramolecular and atomic regrouping and crystallization of the perovskite phase.

Almost up to 700°C there is a gradual destruction of the initial coordination spheres and rearrangement, while the phase composition remains amorphous, but with a progressive increase in the ordering of the atoms. The subsequent temperature elevation in the range of 750-825°C leads to the formation and accumulation of the perovskite crystalline phase in the La_2O_3-Lu_2O_3-Yb_2O_3 system.

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