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Problems of YAG nanopowders compaction for laser ceramics

S.N. Bagayev [a], A.A. Kaminskii [b], Yu.L. Kopylov [c], V.B. Kravchenko [c,*]

[a] Institute of Laser Physics, Russian Academy of Sciences, 630090 Novosibirsk, Russia
[b] Institute of Crystallography, Russian Academy of Sciences, 119333 Moscow, Russia
[c] Institute of Radioengineering and Electronics named after V.A. Kotelnikov, Russian Academy of Sciences, Fryazino Branch, 141190 Fryazino, Moscow Region, Russia

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ABSTRACT

Slip casting and colloidal slip casting at high pressure of yttrium aluminum garnet powders were investigated. It was found that the presence of residual pores in laser oxide ceramics was determined mainly by big size pores in the compact. The size of pore in compact is critical when it is greater than the mean size of initial particles. It was shown that formation of pores' structure in compact was controlled by appearance of quasi-particles in heavy loaded slurry. Pores concentration is critical for ceramics optical transmittance.

1. Introduction

One of the most critical stages in transparent oxide ceramics processing is compaction of initial powders. The general goal of compaction is conversion of ensemble of free spherical or quasi-spherical particles to the structure with one of the known dense packing. Due to permanent aspiration of nano-sized particles for agglomeration the most preferable method for compaction in this case among well known ones is method which gives possibility for self-organization during the packing process. One of these methods is slip casting. Application of this method determined impressing success in laser ceramics processing [1,2]. In spite of rich history of this technology its application for nano-sized powder was not yet investigated in detail. Recently the role of particles' size [3,4], deflocculant type [5] and slurry rheology [6,7] for compact formation by slip casting was investigated, and general requirements for compaction conditions in transparent ceramics processing are understood better now. But laser application requires ceramics with residual porosity less than 10^{-3} vol.%, so more detailed investigation of slip casting and related processes seems necessary.

2. Experiment

Powders of undoped and Nd doped yttrium aluminum garnet (YAG) for experiments were produced by co-precipitation reverse type method. As source of Y and Nd the high purity Y and Nd nitrates water solutions and as sources of aluminum the water solutions of aluminum nitrate or $(NH_4)Al(SO_4)_2{\cdot}12H_2O$ (Donetsk's Chemical Plant production) were used. Y and Nd nitrates were prepared by dissolving of high purity Y (99.95%) and Nd (99.99%) oxides (Giridmet's Plant manufactured) in high purity HNO_3 acid. As precipitant the water solutions of ammonium hydroxycarbonate (reagent grade Panreac) were used. Obtained powders of precursors were transformed to YAG by calcination in the temperature range 900–1300 °C. Powder after calcination was ball milled in water suspension. Shape and size of particles were calculated from SEM and surface specific area (SSA) measurements by BET method. Manufacturing of YAG powders by solvothermal method [8] was also used. The slurry for casting was prepared in ball mill with addition of dispersant–deflocculants. As deflocculants the water solutions of ammonium salts of polyacrilic or polymetacrilic acids with molecular weight in the range 300–16,000 were used. Ammonia water with variable pH index in the range 9.5–11.5 was used as dispersing media. Viscosity of slurry was measured by Brookfield DV-II+ viscosimeter with coaxial cylinders. The size of particles in slurry was measured by laser light scattering method (LLS) with "22 MicroTec" Fritsch's instrument. Compaction was produced by high pressure (200 MPa) colloidal slip casting method [9–11] and by conventional slip casting. Sintering of samples was made in vacuum furnace with carbon heater at 1500–1750 °C with keeping time at the highest temperature during 2–20 h. Pressure in vacuum furnace during sintering cycle was in the range 10^{-4}–10^{-5} Pa. Apparent density of compact and sintered samples was measured by Archimedes's method. Relative porosity of samples sintered at different temperatures was investigated by SEM, AFM and optical microscopy.

* Corresponding author. Address: FIRE RAS, Vvedenskogo squ. 1, Lab. 219, 141190 Fryazino, Moscow Region, Russia. Tel.: +7 903 220 51 18; fax: +7 495 702 95 72.
E-mail address: vbk219@ire216.msk.su (V.B. Kravchenko).

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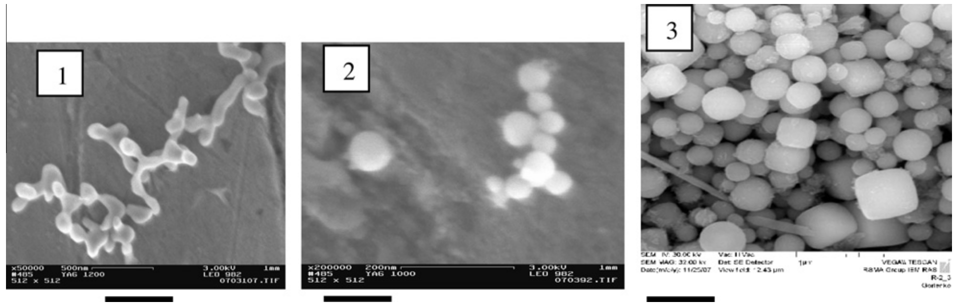

Fig. 1. Nano-sized particles of YAG. 1 and 2 particles produced by co-precipitation method, 1 – hardly and 2 – weekly agglomerated particles, 3 – non-agglomerated particles produced by solvothermal method. Length of bar is 200 nm.

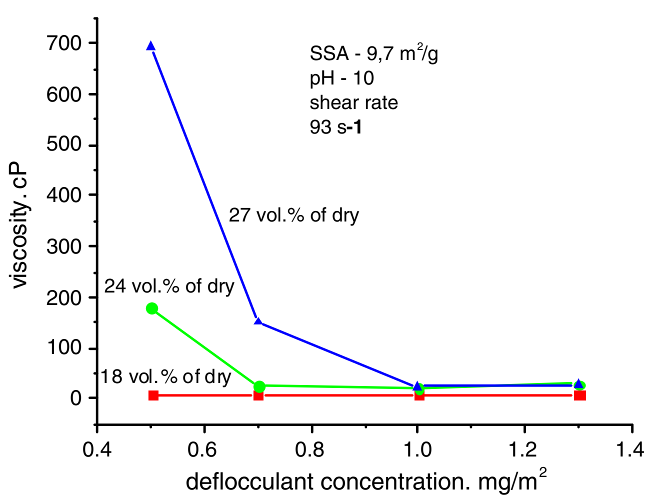

Fig. 2. Viscosity of YAG slurry vs deflocculant content with molecular mass 4000.

3. Results and discussion

Generally powder particles produced by co-precipitation method are strongly agglomerated. The value of agglomeration estimated by SEM depends on precipitation and calcination conditions. At the optimum conditions it is possible to produce powder in which shape and mean dimension of agglomerates can be reduced by ball milling to the shape and size of initial particles as it is demonstrated in Fig. 1.

It is shown in this figure that particles of powders produced by solvothermal method practically are not agglomerated. The value of agglomeration was measured as ratio of mean particles size estimated by LLS to the size of initial particles estimated from SSA and SEM measurements. The average value of this ratio for powders produced by co-precipitation method was 70 and can be reduced (de-agglomerated) to 1–2 by ball milling if co-precipitation and calcinations procedures were optimized. After such de-agglomeration the powder was used for subsequent compaction.

Deflocculant was added to suspension for slurry preparation as weight amount in mg per square meter of total surface square of powder. The optimum quantity of deflocculants was estimated by minimum of viscosity in dependence on deflocculant concentration as shown in Fig. 2.

Value of pH index of slurry for all experiments was chosen as 10.5 because the minimum of zeta potential was found at this pH value. Results of viscosity measurements in dependence on deflocculant molecular weight and powder loading are presented in Fig. 3.

As it is shown in insert of Fig. 3 the powder loading of slurry with viscosity level suitable for slip casting is increased when powder SSA and molecular weight of deflocculant decrease. That means it is preferable to have bigger particle size and smaller molecular weight of deflocculant for heavy loaded slurry.

Viscosity of slurry can be considered as measure of interaction between particles. We divide all possible range of particles

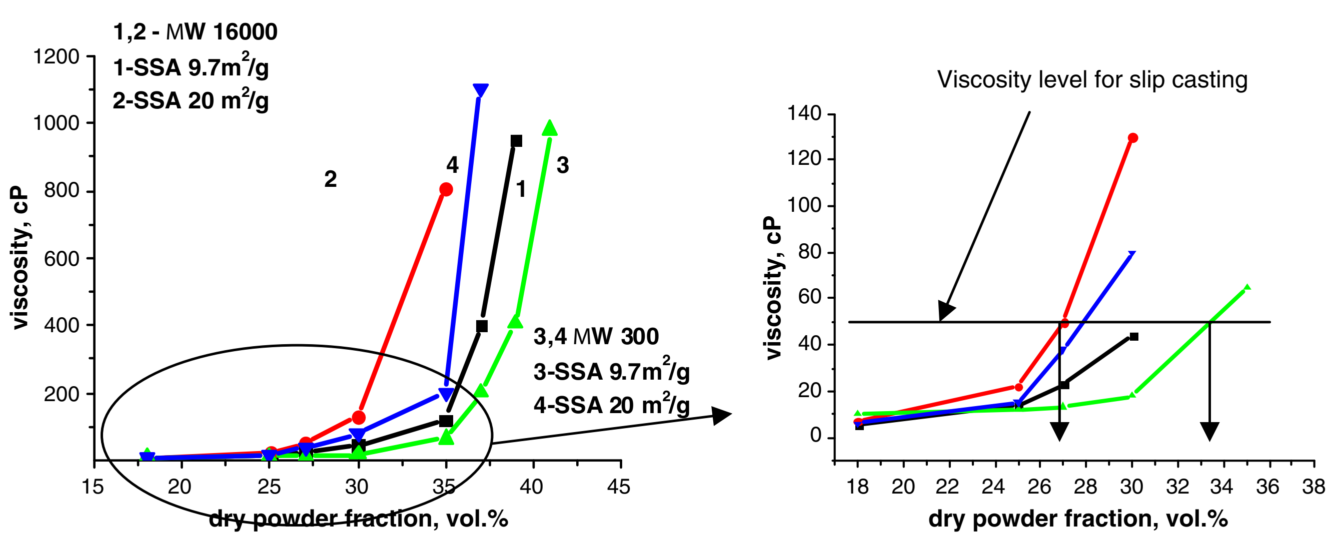

Fig. 3. Viscosity of YAG slurry vs powder fraction for different deflocculants and powders. In the right side of the figure the level of viscosity acceptable for slip casting is fixed and acceptable values of slurry loading are determined.