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PRELIMINARY STUDY IN PREPARATION OF Nd^{3+}: YAG AND Sm^{3+}: Y_2O_3 TRANSPARENT CERAMICS

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Abstract
In the present work we present preliminary research on producing of transparent polycrystalline ceramics. Samarium doped Yttrium oxide (1%, 3% and 5%) and Neodymium (1%) doped YAG ($Y_3Al_5O_{12}$) translucent ceramics were fabricated by solid-state reaction. Commercial nanopowders was used as the starting materials: Sm_2O_3 (6μm) and Y_2O_2 (20-50 nm) for Sm^{3+}: Y_2O_3 and α-Al_2O_3, Y_2O_3 and Nd_2O_3 (20- 50 nm) for Nd^{3+}: YAG. For the preparation of Nd^{3+}: YAG, 0.5 wt% tetraethyl orthosilicate (TEOS) was use as sintering additive, and 1 wt % PEG (polyethylene glycol-400) as dispersant.

1. Introduction

Cubic YAG ($Y_3Al_5O_{12}$) and Y_2O_3 crystals has been investigated for a long time as a laser-host materials due to their high thermal conductivity, broad spectral region, chemical stability, strong Stark-splitting and relatively low phonon energies. However, it is very difficult to grow large size single crystal with high quality because of their high melting point (~ 2430^0C for Y_2O_3 and ~ 1950^0C for YAG). Transparent polycrystalline materials are very good alternative to single crystals because of their sintering temperature, which is much lower than melting point, possibility of having much more active ions, larger size in comparing with single crystal. Since 1995, polycrystalline ceramic laser materials have attracted much attention because the optical quality has been improved greatly and highly efficient laser output could be obtained whose efficiencies are comparable or superior to those of single crystals. In the present work, translucent ceramics was fabricated by a simple solid-state reaction and air sintering. The micro structural properties of Nd^{3+}: YAG and Sm^{3+}: Y_2O_3 ceramics obtained were investigated.

2. Method and samples

We used classical ceramic way (solid state reaction process) for producing the samples. High purity powders of α-Al_2O_3, 99.99+%, ~100 nm (Inframat Advanced Materials LLC USA); Y_2O_3, 99.99%, ~20-40 nm (Nanostructured & Amorphous Materials Inc. USA); Nd_2O_3, 99.95%, ~50 nm (PANGEA INTERNATIONAL LIMITED CHINA) and Sm_2O_3, 99.999%, 3-7 µm (Metall Rare Earth Limited CHINA) were used as starting materials to prepare all the samples.

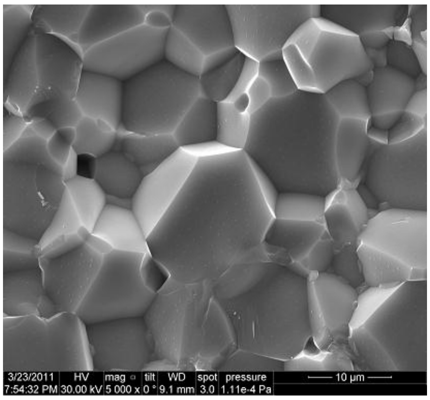
For producing Nd^{3+}: YAG ceramic [1], powders was mixed in stoichiometric ratio with ball mill in anhydrous ethylic alcohol, using alumina balls in an alumina jar, for 48h. As sintering additive 0.5 wt% of tetraethyl orthosilicate (TEOS) is used and as dispersant, in last 2 hours of milling, 1 wt% PEG- 400 (polyethylene glycol- 400) in all sintered samples. Then, the alcohol solvent was removed by spray drying the milled slurry with Buchi Mini Spray Dryer B-290, in nitrogen atmosphere [2]. The parameters for spraying were: inlet temperature 70^0C, feed rate 3ml/min, spray air flow 450l/h and aspiration rate 100%. The spray - dried powder was pressed with low pressure (10MPa) into pellets with half inch diameter in a metal mold and then cold isostatically pressed at 200 MPa. The samples was putted in alumina crucible, surrounded by their own powder and covered with alumina plate. Before sintering the samples are heated at 800^0C for removing organic substances used in preparation. Translucent ceramics were obtained by sintering 16h at 1730°C in air atmosphere, with a growing rate of the temperature by 60^0C/h and a cooling rate of 40^0C/h.

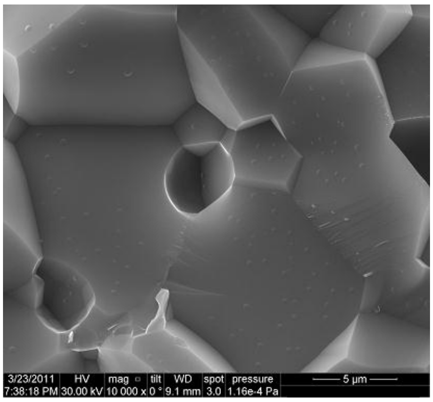
In the case of Sm^{3+}: Y_2O_3 [3, 4], the powders was mixed by stirring in an alumina jar for 48 hours in anhydrous ethylic alcohol. Then, the alcohol solvent was removed by spray drying the milled slurry with Buchi Mini Spray Dryer B-290, in nitrogen atmosphere. The parameters for spraying were: inlet temperature 80^0C, feed rate 3ml/min, spray air flow 450l/h and aspiration rate 90%. The spray- dried powder was pressed with low pressure (10 MPa) into pellets with half inch diameter in a metal mold. The samples was putted in alumina crucible, surrounded by their own powder and covered with alumina plate. Translucent ceramics were obtained by sintering 10h at 1550^0C in air atmosphere, with a growing and cooling rate of 4.3^0C/min.

3. Results and Discussions

A. SEM investigation

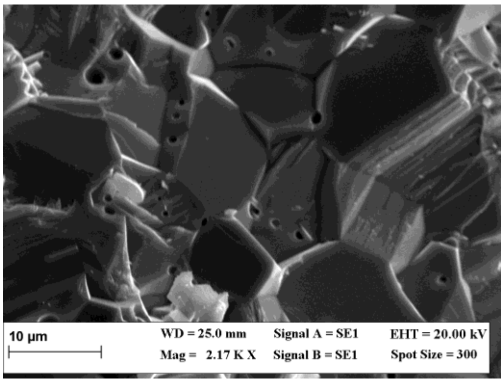
Investigations by electronic microscopy were made for morphological and structural characterization of translucent ceramic samples. SEM investigation of Nd^{3+}: YAG revealed that the average of the grain sizes in ceramics is between 5 and 15 μm (Figs. 1 and 2). They are well packaged, with a few pores due to air atmosphere used during the sintering process.

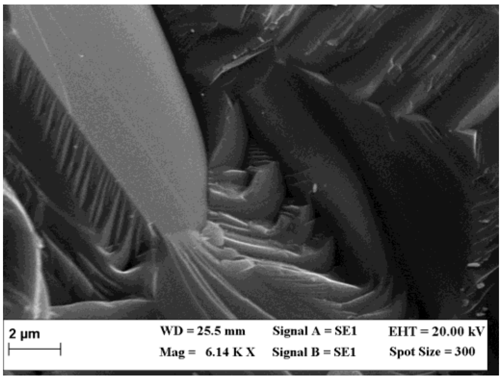




Figs. 1 and 2. SEM micrographs of fracture of 1% Nd:YAG sintered at 1730^{0}C.

In the case of Sm^{3+}: Y_2O_3, details about the stratification and polycrystalline structure in various sections of the samples were carried out (Figs. 3 to 8). The average of grain sizes is between 8 and 12 μm. Because the ceramics were sintered in air atmosphere, they have many pores but they are very well packed.





Figs. 3 and 4. SEM micrographs of fracture of (1%) Sm: Y_2O_3

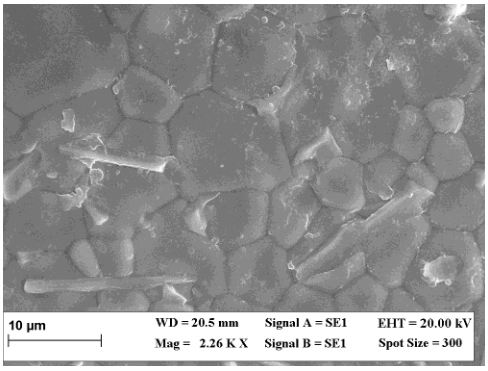


Fig. 5. SEM micrograph of surface of (3%) Sm: Y_2O_3

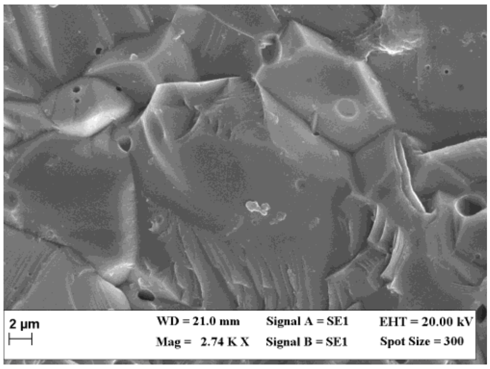


Fig. 6. SEM micrograph of fracture of (3%) Sm: Y_2O_3

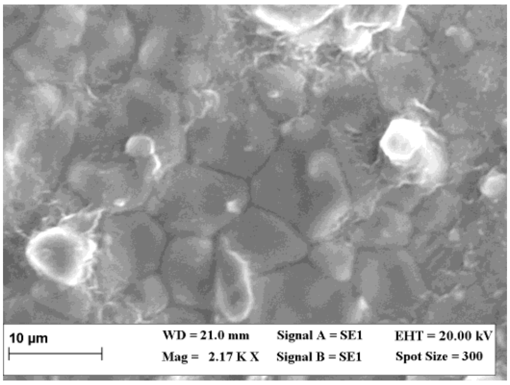


Fig. 7. SEM micrograph of surface of (5%) Sm: Y_2O_3

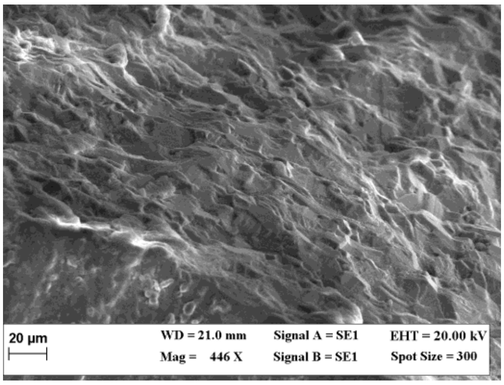


Fig. 8. SEM micrograph of fracture of (5%) Sm: Y_2O_3

B. X-Ray diffraction

X-Ray diffraction experiments were made on Y_2O_3 ceramic powders, with TUR M-62 Diffractometer, using Kα radiation of Cu (λ =1.54051 Å) connected to PC. The obtained diffraction peaks (Fig. 9) correspond to the peak positions of Bragg reflections of Y_2O_3 cubic phase. The insertion of Sm^{3+} ions into the crystalline lattice of the Y_2O_3 ceramics is demonstrated by the diffraction peaks shifting to smaller angles with increasing of Sm content.

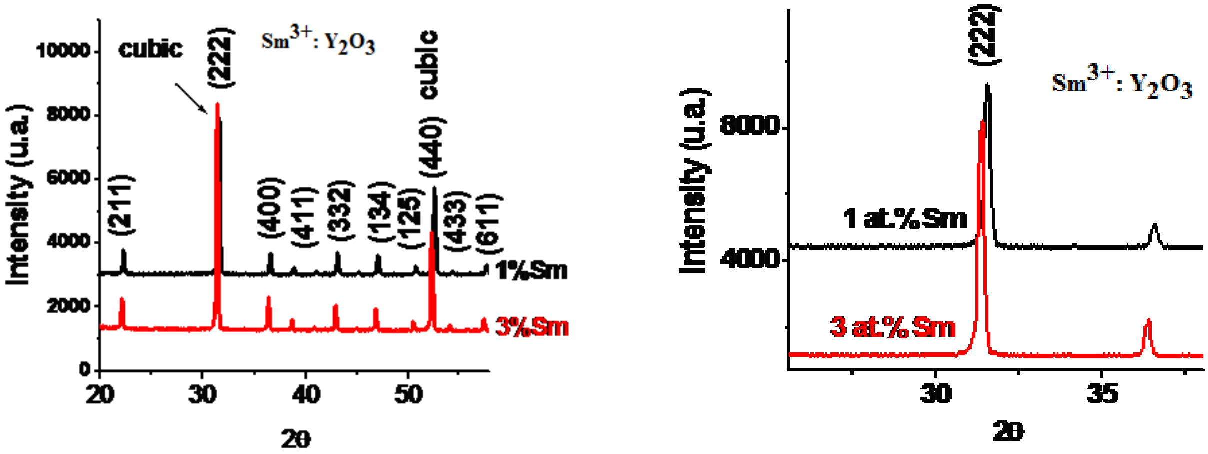


Fig. 9. X-Ray diffraction patterns at 300K on translucent ceramics powders of Y_2O_3: Sm (1%, 3%)

Conclusions

In this work we obtained translucent ceramics using ceramics technology. SEM and XRD experiments showed a well package of constituents and the formation of the cubic phase of ceramics. In our future work, we'll try to obtain transparent ceramics, using vacuum technology.

Acknowledgements

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