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Lead Beneficiary:

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Deliverable Nature	
R = Report, P = Prototype, D = Demonstrator, O = Other	R
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PU = Public	
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A. Abstract / Executive Summary

The activity was concentrated on realizing Nd:YAG poly-crystalline (ceramic) media with improved transparency in comparison with previous obtained samples. Also, composite Nd:YAG structures were fabricated.

B. Deliverable Report

1 Introduction

Ceramic laser materials are now recognized as alternative solutions to single-crystals for realizing lasers with high output performances in free generation mode, in Q-switched regime, or for the generation of sub-ps- or fs- laser pulses. Rare-earth ions-doped cubic or disordered media (that can be also obtained by ceramic methods) may have wide fluorescence bands, suitable for generation of laser pulses close to 100-fs duration.

2 Work performed / results / description

A conventional solid-state reaction was used to prepare the bulk ceramics with compositions of $Y_3AI_5O_{12}$ and Nd^{3+} doped $Y_3AI_5O_{12}$ as well as $Y_{2.985}Nd_{0.015}AI_5O_{12}$ (0.5-at.% Nd), $Y_{2.97}Nd_{0.03}AI_5O_{12}$ (1.0-at.% Nd) and $Y_{2.955}Nd_{0.045}AI_5O_{12}$ (1.5-at.% Nd) as shown in Fig. 1. Powders of AI_2O_3 , Y_2O_3 si Nd_2O_3 with high purity (99,999%) and grains diameter bewteen 50 and 100 nm were used. Based on previous results, polyethylene glycol (PEG-400) was added during the mixing process in order to avoid clustering; also, tetraethyl orthosilicate (TEOS) and MgO were used as sintering additives, in order to obtain good densification by decreasing the compound porosity.

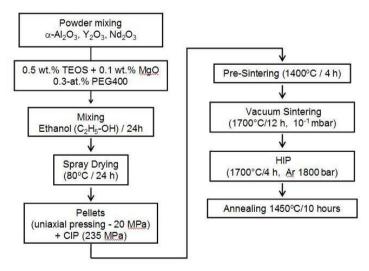


Fig. 1 Flowchart of Nd:YAG ceramics preparation.

Shaping of the dried powder was made in cylindrical pallets of 4.0-mm thickness and 12.0-mm diameter. Besides the samples doped uniformly, composite samples were also prepared. The compositions of the samples with gradual doping were: a) undoped YAG(1.4 mm thickness) + 0.5-at.% Nd:YAG (1.4 mm thickness) + 1.0-at.% YAG (1.4 mm thickness); b) undoped YAG (1.4 mm thickness) + 1.0-at.% Nd:YAG (1.4 mm thickness) + undoped YAG (1.4 mm thickness) + 1.0-at.% Nd:YAG (1.4 mm thickness) + undoped YAG (1.4 mm thickness). Following the pre-sintering in air at 1400°C/4 h, the undoped Y₃Al₅O₁₂ was sintered in vacuum (10⁻¹ mbar) at 1700°C for 12 h; the composite ceramics were presintered in air at 1400°C for 4 h. Next, hot isostatic pressing (HIP) was performed at 1700°C/4 h using Ar gas at 1800 bar pressure for all samples Finally, annealing was made at 1450°C/10 h.

The structure of each sample was investigated with an X-ray diffractometer (PANalytical X'Pert PRO MRD, θ -2 θ geometry, Bragg Brentano). An electronic microscope (SEM, Quanta Inspect F) was used to investigate the morphology (in fracture) of sintered samples.

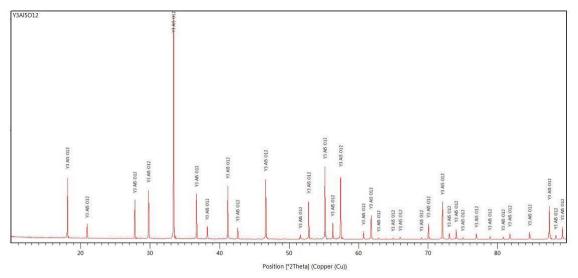


Fig. 2 XRD pattern of Y2.97Nd0.03Al5O12 (1.0-at.% Nd) ceramic.

The XRD pattern for 1.0-at.% Nd:YAG is shown in Fig. 2. All the diffraction peaks were attributed to the cubic symmetry of YAG (*Ia3d* spatial group); the results are similar for all samples, indicating a single phase of $Y_3AI_5O_{12}$.

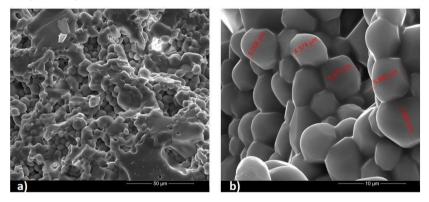


Fig. 3 SEM images of undoped YAG ceramic: a) general view and b) the grain dimensions are shown.

The SEM images of the fracture surface of the undoped YAG is presented in Fig. 3. The structure is inhomogeneous cu intergranular and intragranular porosity (Fig. 3a). The average grain size ranges from 4.37 μ m to 7.44 μ m (Fig. 3b).

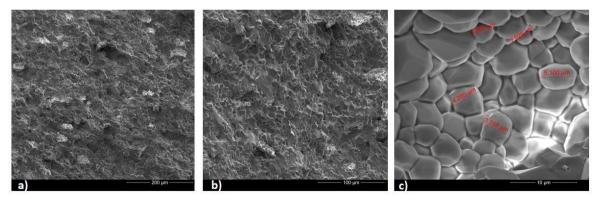


Fig. 4 SEM images of the composite YAG-0.5-at.% Nd:YAG-1.0-at.% YAG ceramic : a), b) general images; c) the grain dimensions are shown.

In the case of the composite YAG-0.5-at.% Nd:YAG-1.0-at.% Nd:YAG ceramic, SEM images reveal a homogenous structure with high degree of densification (Fig. 4a, b). The dimensions of the grains are between 2 μ m and 5 μ m (Fig. 4c). For the composite YAG-1.0-at.% Nd:YAG-YAG ceramic (Fig. 5) the grain size were between 2.49 μ m and 4.5 μ m.

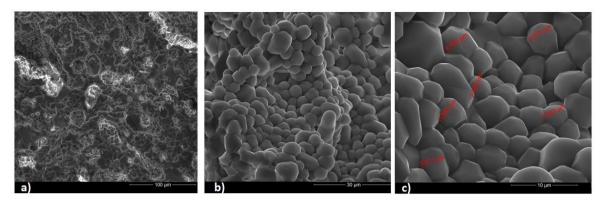


Fig. 5 SEM images of the composite YAG–0.5-at.% Nd:YAG–1.0-at.% YAG ceramic : a), b) general images; c) the grain dimensions are shown.

3 Conclusions

This work has been focussed on improving the quality of Nd:YAG ceramic obtained by solid-state reaction method. Composite structures, made of undoped YAG and Nd:YAG sections with various doping level were realized. The XRD results showed diffraction peaks that were attributed only to the cubic symmetry of YAG. Homogenous structures with high degree of densification and average grain size in the 2 μ m to 5 μ m range were obtained.

4 References

- T. Taira, "RE³⁺-Ion-Doped YAG Ceramic Lasers," IEEE J. of Sel. Top. Quantum Electron., **13** (3), 798-809 (2007).
- Y. Takagimi and Y. Hideki "Rare Earth Garnet Sintered Compact," United States Patent 7,253,129 (2007).
 M.S. Tsai, W.C. Fu, W.C. Wu, C.H. Chen, C.H. Yang, "Effect of the aluminum source on the formation of
- W.B. Liu et al., "Influence of heating rate on optical properties of Nd:YAG laser ceramic," Ceram. Int. 36
- J. Li, W.S. Wu, Y.B. Pan, W.B. Liu, L.P. Huang, J.K. Guo, "Fabrication, microstructure and properties of
- J. Li, W.S. Wu, Y.B. Pan, W.B. Liu, L.P. Huang, J.K. Guo, "Fabrication, microstructure and properties of highly transparent Nd:YAG laser ceramics," Opt. Mater. **31**, 6-17 (2008).
- A. Ikesue and K. Yoshida, "Influence of pore volume on laser performance of Nd:YAG ceramics," J. Mater. Sci. **34** 1189-1195 (1999).
- W. Liu, W. Zhang, J. Li, H. Kou, D. Zhang, Y. Pan, "Synthesis of Nd:YAG powders leading to transparent ceramics: the effect of MgO dopant," J. Eur. Ceram. Soc. **31**, 653-657 (2011).
- A. Ikesue, K. Kamata, K. Yoshita, "Effects of Neodymium Concentration on Optical Characteristics of Polycrystalline Nd:YAG Laser materials," J. Am. Ceram. Soc. 79(7), 1921-1926 (1996).
- A. Ikesue, K. Kamata, "Role of Si on Nd solid-solution of YAG ceramics," J. Ceram. Soc. Jpn. **103**(5), 489-493 (1995).
- Y. Li, S. Zhou, H. Lin, X. Hou, W. Li, H. Teng, T. Jia, "Fabrication of Nd:YAG transparent ceramics with TEOS, MgO and compound additives as sintering aids," J. Alloys & Comp. **502**, 225-230 (2010).
- J. Sanghera, W. Kim, G. Villalobos, B. Shaw, C. Baker, J. Frantz, B. Sadowski, I. Aggarwal, "Ceramic Laser Materials," Materials **5**(2), 258-277 (2012).