

Synthesis and characterization of optical nanocrystals. A step forward towards new promising transparent nanoceramics

M. Galceran*, M. C. Pujol, J. J. Carvajal, X. Mateos, M. Aguiló and F. Díaz

¹*Física i Cristal·lografia de Materials i Nanomaterials (FiCMA-FiCMA). Universitat Rovira i Virgili. Campus Sescelades, c/ Marcel·lí Domingo, s/n, E-43007 Tarragona, Spain
montserrat.galceran@urv.cat

Nanocrystalline materials are polycrystalline materials with a particle size in the nanometer range that have different physical, optical, electronic, chemical and structural properties, because they have a larger fraction of surface atoms than larger-scale materials [1]. These nanocrystals could be the first step for preparing nanoceramic laser material because of the enhancement of the sintering activity due to the nanometric size dimension [2]. Nanoceramics are very attractive as solid state lasers due to their several advantages respect to the bulk single crystal, such as low cost, easy fabrication and good mechanical and optical properties. Furthermore, ceramics significantly improve the thermal shock parameter and resistance to laser damage allowing high power laser operation [3].

Since 1998, highly transparent ceramic lasers have been fabricated by vacuum sintering, using nanocrystalline materials such as Nd:Y₃Al₅O₁₂ (Nd:YAG)[4], Nd:Lu₂O₃ [5], Yb:Sc₂O₃ [6], and Yb:Lu₂O₃ [7]. Recently, the high-pressure low-temperature technique (HPLT) has emerged to obtain transparent ceramic lasers such as YAG [8] and RE:YAG [9,10]. The most outstanding advantage of the HPLT method, with respect to other techniques, is the low sintering temperature required to prepare the ceramic material. In addition, it is a new, non-conventional method for avoiding grain growth during sintering.

KRE(WO₄)₂ (RE = Eu³⁺, Gd³⁺, Er³⁺ and Yb³⁺), RE₂O₃ (RE = Sc³⁺, Ho³⁺, Er³⁺, Yb³⁺ and Lu³⁺) nanocrystals for solid state laser applications were synthesized by the modified Pechini method [11,12,13] (figure 1). The Pechini method is an alternative sol-gel technology used to synthesize nanocrystals [14]. The sol-gel process offers several advantages, such as low cost, versatility, simplicity, low processing temperature and a high degree of homogeneity. The experimental variables were optimized as a function of the desired material to synthesize. The synthesis of nanocrystals is the starting point to obtain ceramic materials using the high pressure low temperature method (figure 2).



Fig. 1: Nanocrystals synthesized using the modified Pechini method.

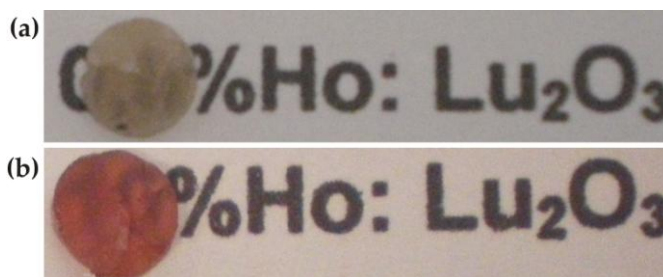


Fig. 2: (a) 0.5% at. Ho:Lu₂O₃ and (b) 10% at. Ho:Lu₂O₃ nanoceramics.

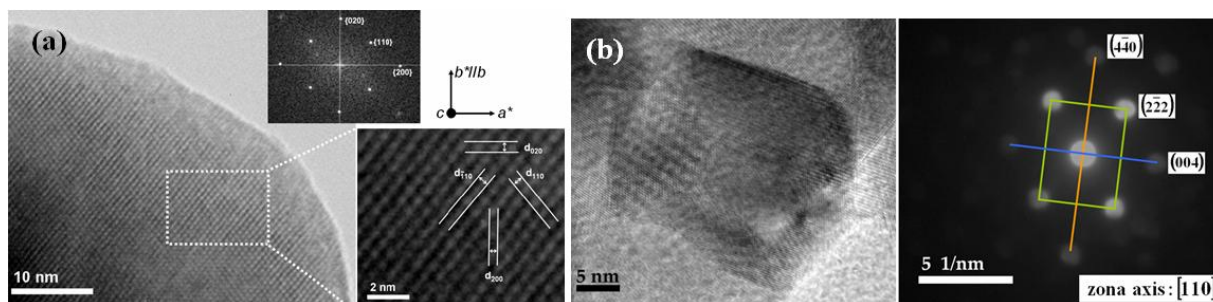


Fig. 3: HRTEM image and electron diffraction analyses of (a) KREW nanocrystals and (b) RE₂O₃ nanocrystals

The structural characterization of nanocrystals was investigated by a number of techniques, the most common being X-ray diffraction and electron diffraction. Figure 3 shows an example of high resolution

transmission electronic microscopy of the oxide nanocrystals, where it's possible to observe the lattice plane fringes indicating the high degree of crystallinity. The Fast Fourier Transform pattern and the electron diffraction pattern were used to index the lattice planes in accordance to their crystalline phase. On the other hand, the morphology, the average particle size and the particle size distributions were carried out using electronic microscopy (figure 4). Moreover, the optical absorption and luminescence measurements were performed at room and low temperature in order to determine the energy levels of the electronic states.

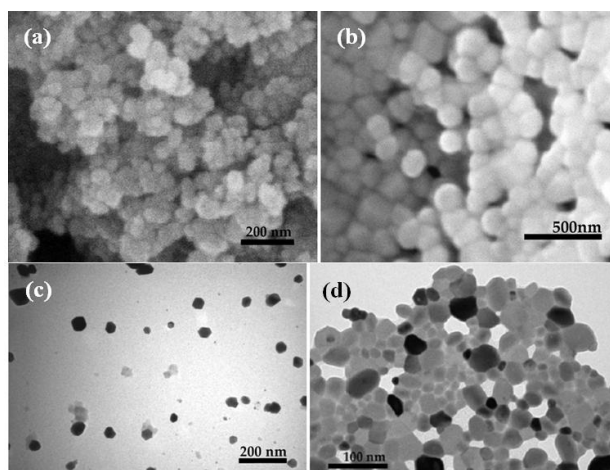


Fig. 4: SEM image of (a) KREW nanocrystals and (b) RE_2O_3 nanocrystals and TEM images of (c) KREW nanocrystals and (d) RE_2O_3 nanocrystals

References

- [1] G. Cao, "Nanostructures and Nanomaterials - Synthesis, Properties and Applications", Imperial College Press (2004).
- [2] C. Liu, X. Wang, Y. Jiang, Y. Wang and S. Hao, *Rare Metals*, **25** (2006), 471
- [3] A. Ikesue, Y. L. Aung, *Nature Photonics* **2** (2008) 721
- [4] J. Lu, K. Ueda, H. Yagi, T. Yanagitani, Y. Akiyama and A. A. Kaminskii, *Journal of Alloys and Compounds* **341** (2002) 220
- [5] J. Lu, K. Takaichi, T. Uematsu, A. Shirakawa, M. Musha, K. Ueda, H. Yagi, T. Yanagitani and A. A. Kaminskii, *Applied Physics Letters* **81** (2002) 4324
- [6] J. Lu, J. F. Bisson, K. Takaichi, T. Uematsu, A. Shirakawa, M. Musha, K. Ueda, H. Yagi, T. Yanagitani and A. A. Kaminskii, *Applied Physics Letters* **83** (2003) 1101.
- [7] K. Takaichi, H. Yagi, A. Shirakawa, K. Ueda, S. Hosokawa, T. Yanagitani and A. A. Kaminskii, *Physica. Status Solidi A* **202** (2005) R1
- [8] D. Hreniak and W. Streck, *Journal of Alloys and Compounds* **341** (2002) 183-186
- [9] R. Fedyk, D. Hreniak, W. Lojkowski, W. Streck, H. Matysiak, E. Grzanka, S. Gierlotka and P. Mazur, *Optical Materials* **29**, 1252-1257 (2007).
- [10] W. Streck, A. Bednarkiewicz, D. Hreniak, P. Mazur and W. Lojkowski, *Journal of Luminescence* **122-123** (2007) 70
- [11] M. Galceran, M. C. Pujol, M. Aguiló and F. Díaz, *Journal of Sol-Gel Science and Technology* **42** (2007) 79
- [12] M. Galceran, M. C. Pujol, M. Aguiló and F. Díaz, *Materials Science and Engineering B* **146** (2008) 7
- [13] M. Galceran, M. C. Pujol, M. Aguiló and F. Díaz *Journal Physical Chemistry C* **113** (2009) 15497.
- [14] M. P. Pechini, US Patent 3330697 (1967)