

## II.7.14

**Synthesis of KY(WO<sub>4</sub>)<sub>2</sub> and Doped Nanocomposites by Complex Sol-Gel Process.** Andrzej Deptuła<sup>1</sup>, M. T. Borowiec<sup>2</sup>, W. Lada<sup>1</sup>, T. Olczak<sup>1</sup>, D. Wawszczak<sup>1</sup>, T. Zayarniuk<sup>2</sup>, V. Domukhovskii<sup>2</sup>, P. Aleshkevych<sup>1</sup> and H. Szymczak<sup>2</sup>; <sup>1</sup>Institute of Nuclear Chemistry and Technology, Warsaw, Poland; <sup>2</sup>Institute of Physics, Polish Academy of Sciences, Warsaw, Poland.

A complex sol-gel process and use of ascorbic acid as a chelating agent were applied to preparation of KY(WO<sub>4</sub>)<sub>2</sub> and nanocomposites of KY(WO<sub>4</sub>)<sub>2</sub> doped with 1% additive. The rare-earth double tungstate compounds ARe(WO<sub>4</sub>)<sub>2</sub>, where A = K,Rb and Re = Dy, Er, Ho, Nd, Yb, are attractive materials as laser hosts [1] and of special interest in regard to magnetic and structural phase transitions [2]. Saturated tungsten sols (0.15 M) were prepared by dissolving (NH)<sub>10</sub>H<sub>2</sub>(W<sub>2</sub>O<sub>7</sub>)<sub>6</sub> in an aqueous ascorbic acid solution. Y and Y + Yb nitrates were added to this solution. Sols were then gelled under reduced pressure and thermally treated. Thermogravimetric and differential thermal analyses coupled with X-ray diffraction measurements indicated that the final nanocomposites formed at the relatively low temperature of approximately 420C. Carbon-free products were obtained by firing at 550C for 10 h or at 650C for various times. To avoid these drying procedures, self-ignition was induced at temperatures of 250-450C. Only gels with high specific surface areas could be ignited. The decarbonization temperatures of tungstates were reduced from 650C through use of chemical treatment with concentrated nitric acid and hydrogen peroxide. Heat treatment of the resulting slips at 550C for 2 h yielded carbonate-free tungstates. X-ray diffraction (XRD) and electron paramagnetic resonance spectra were studied. The expected monoclinic phase C2/c of the KYW nanocomposites was confirmed by XRD. The unit cell parameters and the sizes of particles were determined. Electron spin resonance studies in the X-band were performed on the KYW and KYW:Yb nanocomposites, and on the KYW single crystals. Additional lines observed for the doped KYW were attributed to the presence of Yb ions. [1] J.A. Caird and S.A. Payne, Handbook of lasers science and technology, ed. M.J. Weber (1991); A.A. Kaminskii, Today and tomorrow of laser crystal physics, Phys. Stat. Sol. A 148 (1) 1995. [2] M.T. Borowiec, The phase transitions in double tungstates - in extremely low-dimensional and low-symmetry compounds with cooperative Jahn-Teller effect, Proc.SPIE 4412 (2001) 196. \*This work was supported in part by the EU project DT-CRYS and by the Polish State Committee on Science (KBN; Project No. 72/E-67/SPB/6.PR/DIE 430/2004-2006).