

## Nanostructure Obtained by Sol-gel Acrylamide Polymerization in Y-V-O System.

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Sol-gel acrylamide polymerization is one of the most promising ways to get an excellent homogenization at an atomic scale of the elements [1]. For that reason the main purpose of this paper is to evaluate the influence acrylamide polymerization on the microstructure to obtain nanocrystals in a *Y-V-O* system.

The Y-V-O system was prepared by sol-gel acrylamide polymerization.  $Y_2O_3$  and  $V_2O_5$  were separately dissolved in  $HNO_3$  with distilled water. Ethylenediamine-tetraacetic acid was used as a chelating agent to isolate *Y* and *V* cations. Then, the two solutions were mixed and  $NH_4OH$  was used to adjust the pH to 3.4. To this solution we added acrylamide,  $C_7H_{10}N_2$ , and a chemical initiator  $C_8H_{18}N_6 \cdot 2HCl$  at  $80^\circ C$  to accomplish the polymerization. This process took 8 min. The obtained gel was dried at  $200^\circ C$  for 12h; it results in a porous foam material (xerogel).

The structure of xerogel was identified using a Bruker X ray powder diffractometer –AXS D8-ADVANCE with  $\lambda(CuK\alpha) = 1.5406\text{\AA}$  radiation. Fig. 1 shows the transformation from the nanocrystals phase to the crystalline phase  $YVO_4$  (JCPDS 70-1281) as a consequence of the increase in the temperature of the xerogel powders. In Fig. 2a one observes a particle size between 30 to 100 nm. These results were obtained by Transmission Electron Microscopy TEM with a JEM – 1200EX microscope.

The xerogel was grinded in an agate mortar, and the powder was annealed first at  $250^\circ C$ . Then we increased the temperature in  $100^\circ C$  steps up to  $800^\circ C$ . In each step the temperature was kept constant for 12h in air atmosphere. The resulting material was compacted into pellet shapes exerting a pressure of  $4\text{ ton/cm}^2$  for 15 min under vacuum. The pellets were then subjected to thermal treatment at  $850^\circ C$  for 4 days in air atmosphere. After this, the sample was reduced with Zr in vacuum at  $850^\circ C$  during 27 days and quenched in ice. Zr was used as a gatherer in order to obtain the crystalline phase  $YVO_3$  (JCPDS 25-1066). Scanning Electron Microscopy SEM and Electron Dispersive X-ray (EDX) were performed on a Leica-Cambridge Stereoscan 440, equipped with an Oxford/Link System electron probe microanalyzer (EPMA). SEM micrographs (Fig. 2b and c) were taken to follow the evolution of crystal growth during five months. The morphology shows an increase of particle size which at the end (Fig. 2c) varies from 1 to  $100\ \mu m$ , it also exhibits uniform grains. The results of the quantitative EDX analysis (Fig. 3.) indicate that the theoretical atomic percentage is in agreement with the measured atomic percentage.

### References

[1] A. Sin, and P. Odier. Adv. Mater. 12, 9, (2000) 649-652.

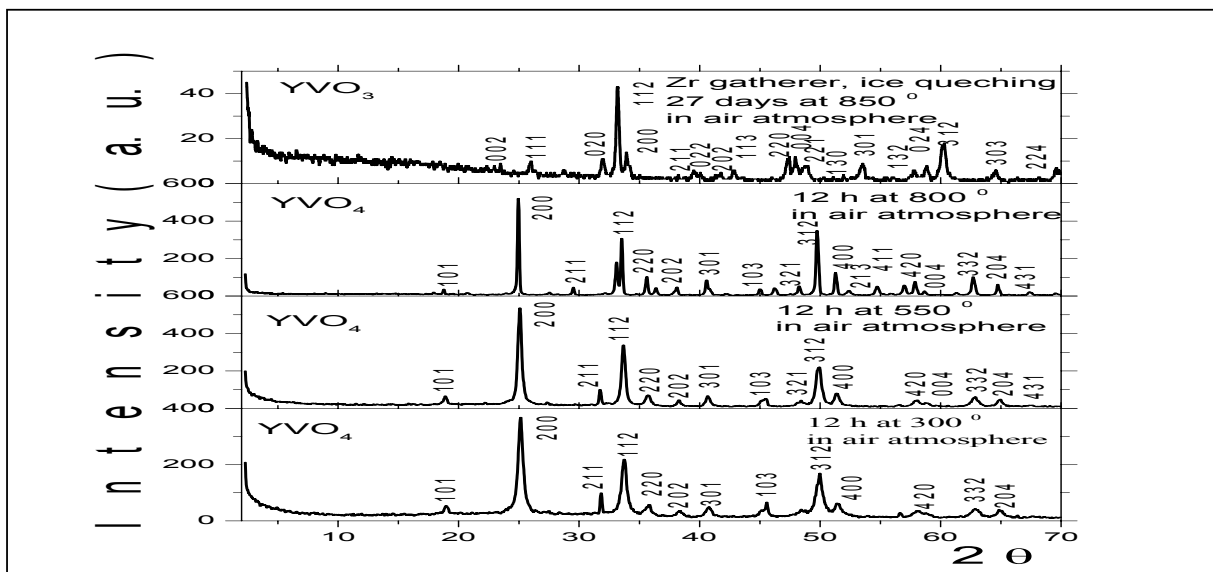


Fig. 1. X-ray pattern diffraction of  $YVO_3$  (up). Evolution of the crystal structure of  $YVO_4$  (down).

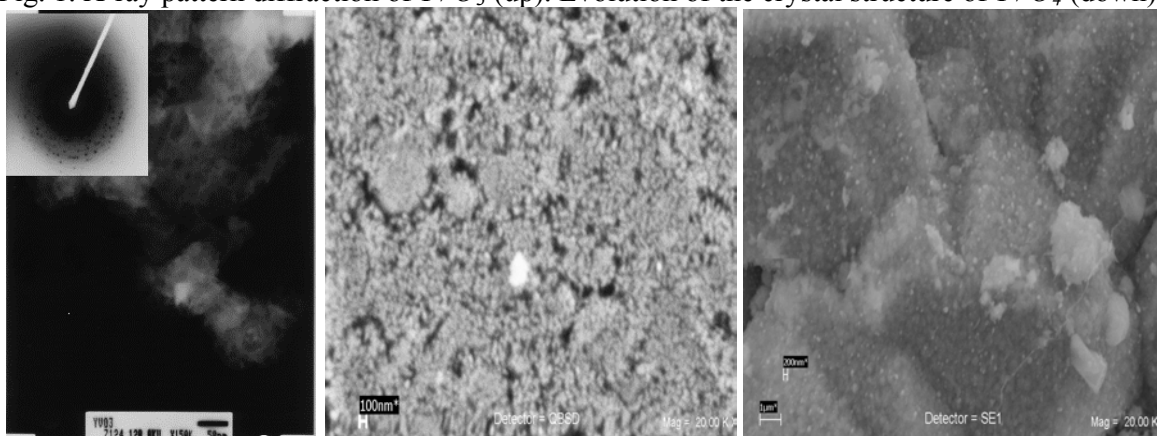


Fig. 2. Diffraction pattern and image TEM associated with xerogel at room temperature after milled (left), c) SEM micrograph taken on the surface of the  $YVO_4$  compound (middle).  $YVO_4$  single crystal phase after reduced with Zr at 5.0Kx (right).

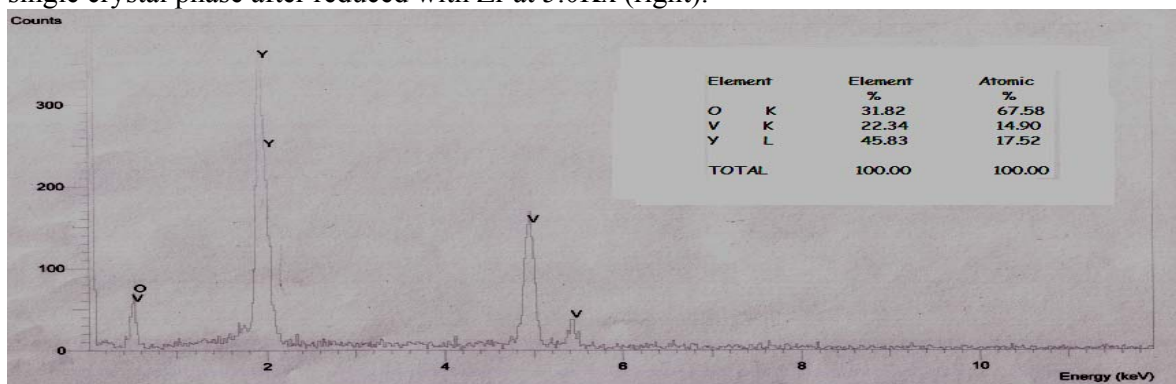


Fig. 3. Energy dispersive x-ray spectra (EDS) from surface of  $YVO_3$  sample. Electron beam accelerating voltage = 20 kV, x ray counting live time = 100s.