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Microstructural Comparison of La-V-O Compounds prepared by Sol-Gel Acrylamide Polymerization and Solid State Reaction.

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$LaVO_4$ and $LaVO_3$ are lanthanum orthovanadates that belong to a group of compounds with interesting structural, electronic, magnetic and electrical properties. The crystal structure of $LaVO_4$ compound has been reported in two polymorphs, namely, tetragonal zircon-type (*t-*$LaVO_4$) isostructural to $ZrSiO_4$ compound and monoclinic monazite-type (*m-*$LaVO_4$). $LaVO_3$ compound has an orthorhombic distorted perovskite structure first found in $GdFeO_3$ compound.
The purpose of this work is to compare the influence of sol-gel acrylamide polymerization synthesis on the crystal structure and microstructure in *m-*$LaVO_4$ and $LaVO_3$. These results were contrasted with the samples obtained by solid state reaction (SSR). The differential thermal analysis (DTA) for SGAP shows the formation of *m-*$LaVO_4$ occurs at 400 ^oC, in comparison with the sample prepared by SSR at 1400 ^oC [1].
Fig. 1 (a) shows the morphology and roughness obtained by atomic force microscopy (AFM). The image reveals needle shape particles (needles are made of metal-EDTA and polymer). This crystallization style depends of EDTA molar concentration and pH value. We use a solution of 1:1 vanadium-EDTA molar ratio and we adjusted the pH to 5.4 with NH_4OH. Because the fast acrylamide polymerization is generally in aqueous medium whose pH is close to neutral [2]. It has been reported that weak ligand such as EDTA adjusts the morphology and uniformity of crystals shape in the crystallization process. Also, Jia *et al.* [3] reported that the pH value ranged from 7-13 exhibited rods like morphology. The needle shape was maintained up to the formation of $LaVO_4$ compound. This result was confirmed by scanning electron microscopy (SEM) micrographs, see Fig. 1 (b). The *m-*$LaVO_4$-SGAP reveals a homogeneous morphology with needle-shaped grains of 50 *nm* average size. The SSR present a broader size distribution in the micrometer range.
Both *m-*$LaVO_4$ samples were reduced into $LaVO_3$ using a *Zr* rod at 850 ^oC in vacuum. Fig. 2 (right) shows a homogeneous grain distribution with an average size of 745 *nm* for $LaVO_3$-SGAP. $LaVO_3$-SSR has an average size of 3.45 μm (Fig. 2, left). The stoichiometry of all compounds was confirmed by energy dispersive X-ray spectroscopy (EDX). X-ray powder diffraction (XRD) and transmission electron microscopy (TEM) give crystal structures in agreement with those reported in the literature. An image from TEM study for $LaVO_3$-SGAP is shown in Fig. 3. The morphology is in agreement with SEM results.

References

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[3] C. Jia, et al., *J. Phys. Chem*. B 109 (2005) 3284.
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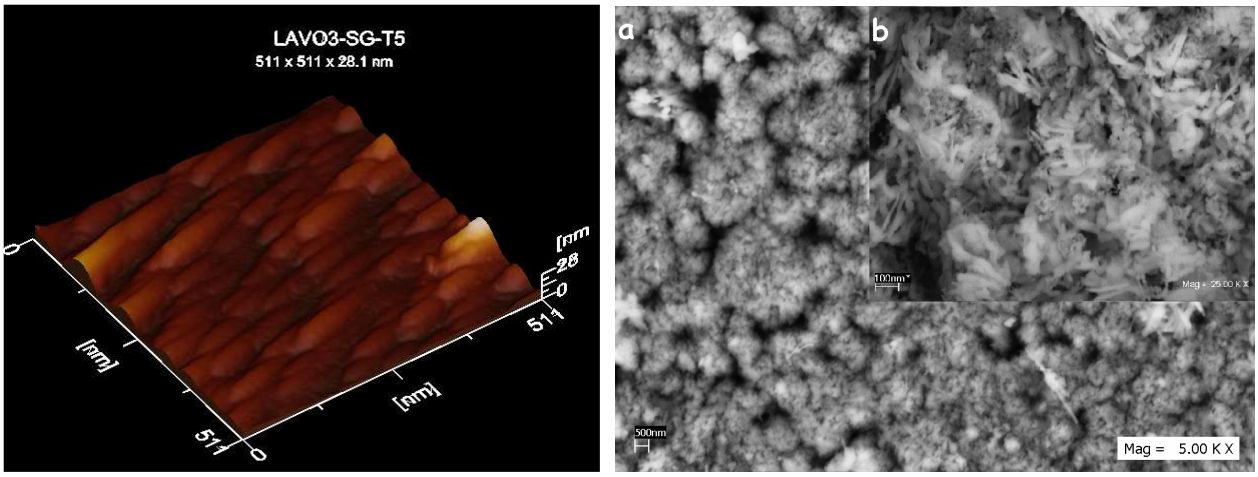

Fig. 1 (a) AFM micrograph obtained by AC mode of gel prepared by SGAP. The image size is 511 x 511 *nm* and clock speed of 666.70 μs. (b) SEM micrograph of *m-*$LaVO_4$-SGPA at the end of synthesis at 400 ^oC during 12 hours in air.

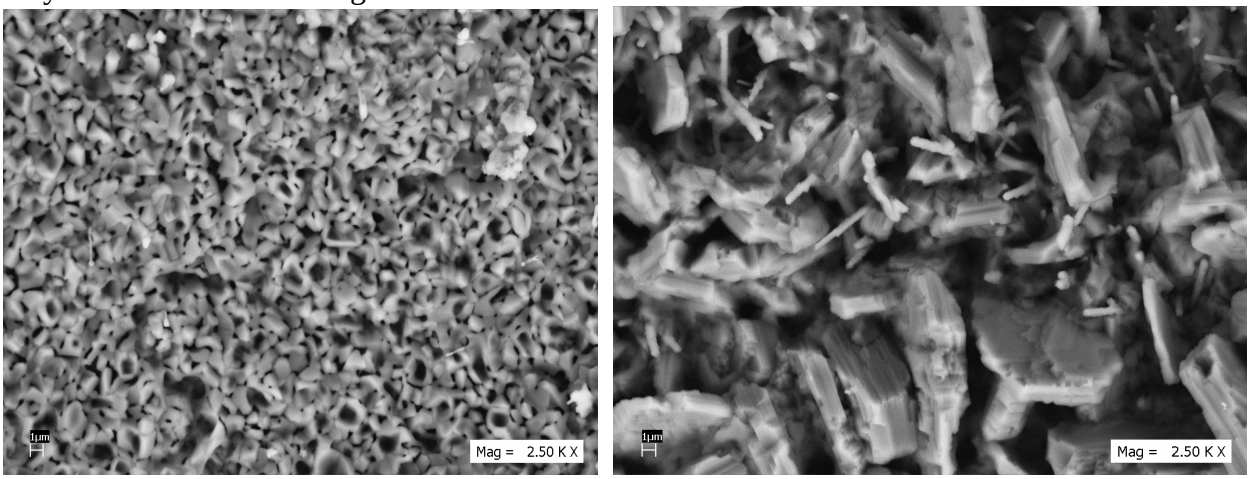

Fig. 2. Comparison of microstructure obtained by SEM on surface of $LaVO_3$ compound at the end of heat treatment at 850 ^oC during 15 days prepared by SGAP (right). SEM micrograph of surface of $LaVO_3$ compound into a pellet topology at 850 ^oC during 15 days prepared by SSR.

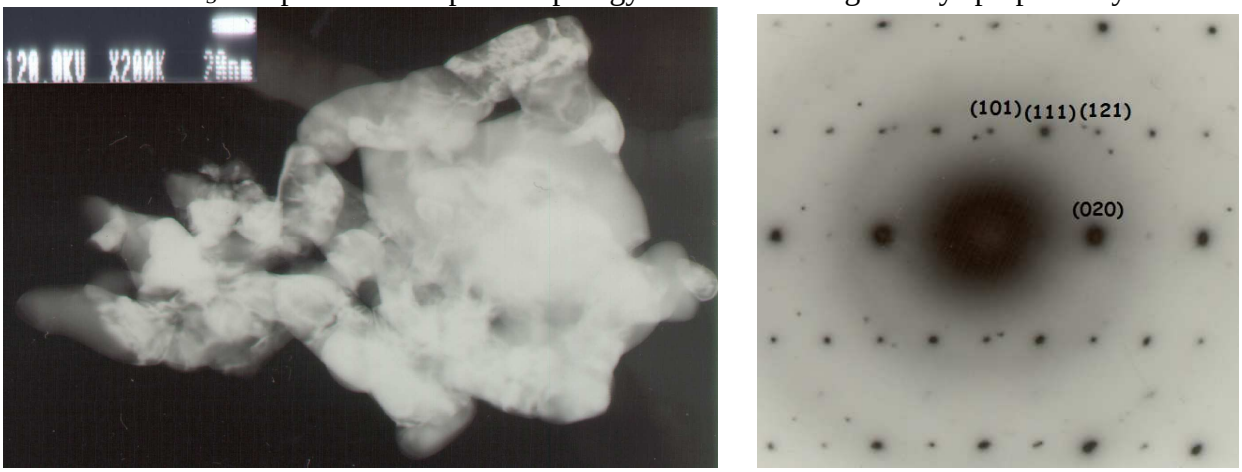

Fig. 3. TEM micrograph powders of $LaVO_3$ compound prepared by SGAP and indexed diffraction pattern of [101] zone axe.

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