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Heat treatment effect on the structure and morphology of strontium monoacid orthophosphate thin films

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Abstract

Highly crystalline β -type strontium hydrogen phosphate (β -SrHPO₄) thin films were prepared by a simple electrodeposition method. Their composition and morphology were investigated via X-ray diffraction (XRD) and a scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy system (EDX). It was found that both the structure and the morphology of the thin films were changed as a reason of a heat treatment at 600°C.

Keywords: Strontium hydrogen orthophosphate; heat treatment; strontium pyrophosphates; morphology change; phase transformation .

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1 Introduction

Strontium has been shown to be a trace element in the human body that stimulates the bones formation. It is used as a kind of industrial chemical and analytical reagent. SrHPO₄ can be used in different fields including luminescent materials, all-solid-state lasers, surface conditioner, ceramics batteries, fuel cells, adsorption of heavy metals and dyes, drug carrying etc. [1–4] Crystallographically, SrHPO₄ has three different types of structures: Triclinic α -SrHPO₄ in which the crystals are built up of SrO₈ square antiprisms and PO₄ tetrahedra which are linked to form a three-dimensional network [5], β -SrHPO₄ with a hexagonal structure and finally γ -SrHPO₄ with an orthorhombic cell and a space group of Pbc_a [6].

In the present study, the strontium hydrogen phosphates thin film was subjected to a heat treatment at 600 °C, the obtained results were discussed in terms of the surface structural change as well as the phase transformation.

2 Experimental Methods

2.1 Reagents

Strontium nitrate (Sr(NO₃)₂), diammonium phosphate ((NH₄)₂HPO₄) were purchased from Fluka with analytical grade. All chemicals were used as received without further purification, and all solutions were prepared with deionized water.

2.2 Materials Preparation

The SrHPO₄ thin films were prepared by a cathodic electrodeposition method as already reported [7,8]. Firstly, solution A was prepared by dissolving Sr(NO₃)₂ in 50 mL deionized water under mechanical stirring for 20 min. Secondly, solution B was prepared by dissolving (NH₄)₂HPO₄ in 50 mL deionized water. Solution B was added into solution A under vigorous mechanical stirring for 10 min. The mixture was then used as the electrolyte to deposit SrHPO₄ using a standard electrochemical assembly with three electrodes (Saturated Calomel Electrode (SCE) as the reference electrode, Platine rod as the counter electrode, and Fluorine-doped tin oxide (FTO) was used as the substrate after it was cleaned ultrasonically by acetone and distilled water. The three electrodes were connected to SP-50 Bio-Logic Science Instruments potentiostat.

The deposition conditions including deposition time, applied current density and initial precursors' concentrations were optimized through different experiments to give an optimum deposition time of 30 minutes, concentration of Strontium nitrates of 4×10^{-2} , phosphate nitrates of 2.67×10^{-2} , and a deposition current density of -1 mA cm^{-2} .

2.3 Characterization

The structure of the samples was characterized by X-ray diffractometer (Bruker D8) using CuK α radiation ($\lambda = 1.5406 \text{ nm}$) with a step rate of 0.025/s over the range of 2 θ from 10 to 70°. The morphology of samples was characterized by scanning electron microscope (Jeol JSM-6460LAV) operating at 15 kV and equipped with an energy-dispersive X-ray spectroscopy system (EDS).

3 Results and Discussion

3.1 Microstructure of β -SrHPO₄ films without heat treatment

Figure 1A shows the XRD pattern of the as-synthesized thin films. All the peaks are consistent with the standard data of β -type SrHPO₄ (JCPDS card No. 12-0368), indicating that the obtained sample had a pure hexagonal crystal structure [2]. The EDS microanalysis shown in **Figure 1B** was used to determine the composition of the electrode. It was confirmed that the following elements are present: Sr, P, Sn and O with experimental atom fractions of 11.85%, 12.13% and 58.10% respectively.

The SEM images (**Figure 2**) show that the obtained sample is composed of nanosheets, which self-assembled as flower-like spherical particles with average size of about 20 μm .

Fig. 1. Characterization of the β -SrHPO₄: (A) XRD pattern, (B) EDXS microanalysis

Fig. 2. SEM micrographs of the hexagonal β -SrHPO₄ obtained at 15 keV primary beam energy.

3.1.1 Microstructure of β -SrHPO₄ thin films with heat treatment at 600 °C

The SrHPO₄ heat-treated at 600 °C was characterized using X-ray diffraction in order to identify the new structure of the thin film, the XRD pattern obtained is presented in Figure 3A, it is found that the phase obtained is a mixture of two strontium pyrophosphate phases (Sr₂P₂O₇) following the JCPDS cards N° 00-012-0367 and N° 01-075-1490. The morphology of sample is shown in Figure 4, one can see that the thin flaky crystals of the Sr₂P₂O₇ phase are damaged and cracks are appeared as a result of the heat treatment leading to adherence degradation. A suitable heat treatment is then required.

Fig. 3. Characterization of the Sr₂P₂O₇: (A) XRD pattern, (B) EDX microanalysis

Fig. 4: SEM images of the Strontium pyrophosphates Sr₂P₂O₇ obtained at 15 keV primary beam energy.

4. Conclusion

In summary, this work provided a simple method to deposit β -SrHPO₄ clusters onto the surface of FTO. β -SrHPO₄ flaky crystals with high purity, good crystallization and uniform morphology could be successfully obtained. XRD and SEM observations revealed that the SrHPO₄, under heat treatment at 600 °C, is transformed to Sr₂P₂O₇ and the film is damaged as shown by the apparition of cracks.

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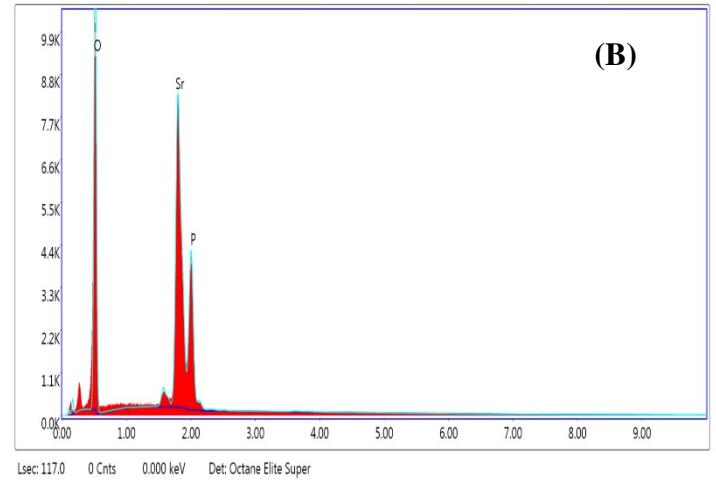
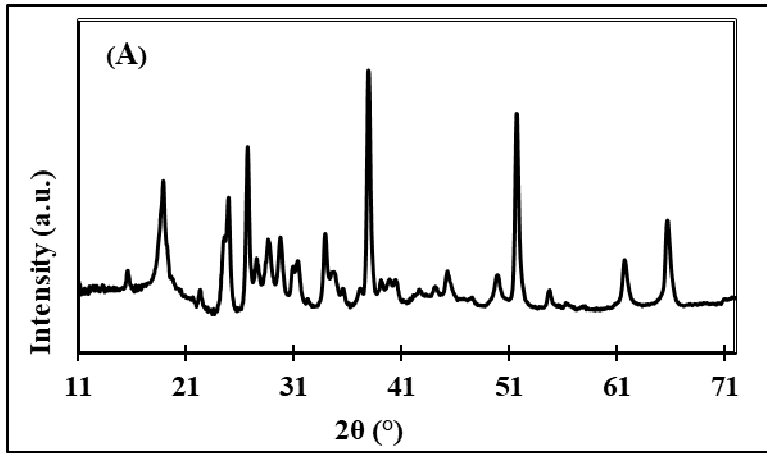


Fig. 1. Characterization of the β -SrHPO₄: (A) XRD pattern, (B) EDXS microanalysis

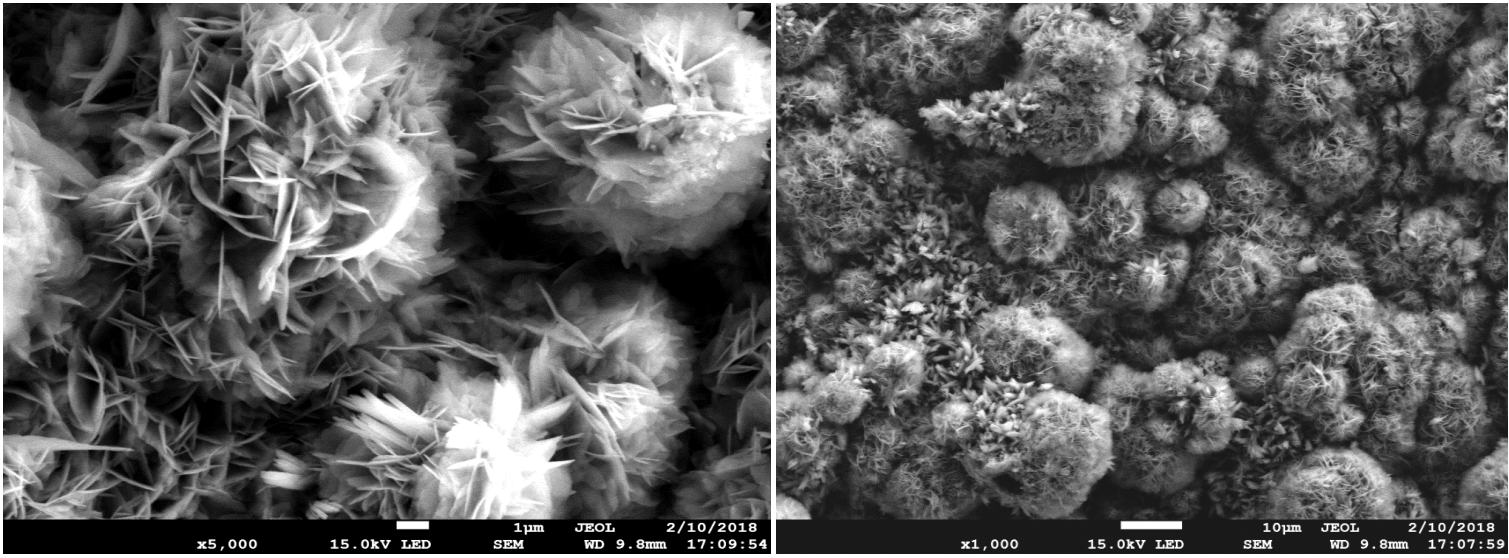


Fig. 2. SEM micrographs of the hexagonal β -SrHPO₄ obtained at 15 keV primary beam energy.

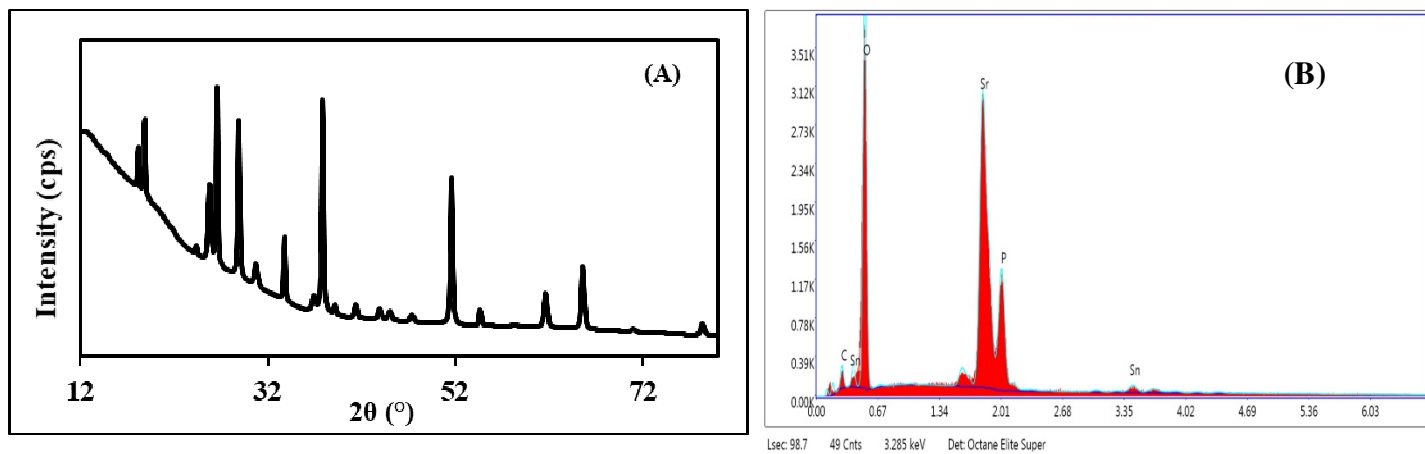


Fig. 3. Characterization of the $\text{Sr}_2\text{P}_2\text{O}_7$: (A) XRD pattern, (B) EDX microanalysis

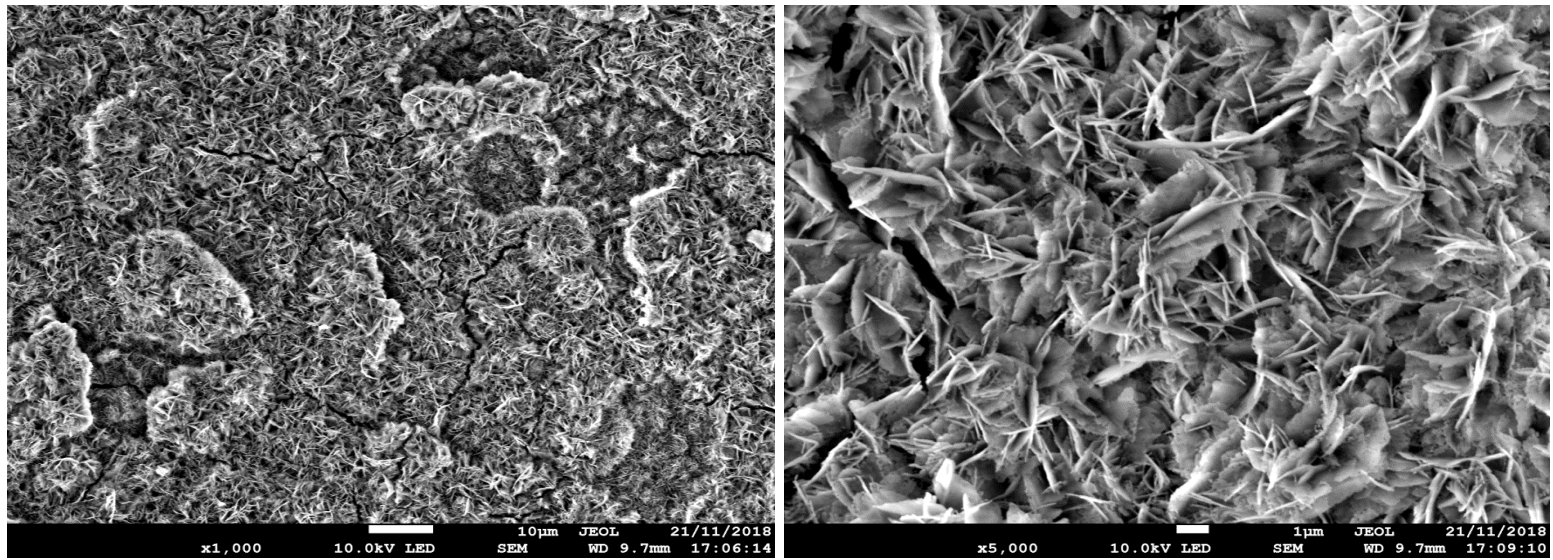


Fig. 4: SEM images of the Strontium pyrophosphates $\text{Sr}_2\text{P}_2\text{O}_7$ obtained at 15 keV primary beam energy.