

NOVEL SYNTHESIS OF SrSe BY REDUCTION OF SELENIUM INTO RONGALITE SOLUTIONS

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In this work a new methodology to synthesize strontium selenide on the base of reduction of selenium is presented. The selenium comes from oxidation stage 0 to -2, afterward it is mixed with a strontium salt, strontium chloride. In this process a fast substitution of chemical reaction is realized. In order to make the optical characterization, the adsorption in the UV-visible range was measured leading to compute direct and indirect band gap energies. The obtained strontium selenide is a black powder dispersed in aqueous solution, the obtained Band gap values were 5.19 and 4.53 eV for direct and indirect respectively. From the Raman spectrum was identified three peaks: at 114 and 234 cm^{-1} corresponding to strontium selenide, and 456 cm^{-1} corresponding to strontium oxide. The infrared response shows Sr^{2+} at 524 cm^{-1} , strontium oxide at 615 cm^{-1} plus O-H vibration and bending vibration H-O-H. Finally from transmission electron microscopy a crystalline structure was found.

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1. Introduction

In the last years, strontium selenide compounds have recently received considerably interest from both theoretical and experimental points of view. Important characteristics ranging from catalysis to microelectronics have been found [1]. In [2] was found in the nonrelativistic regime that the indirect band gap of SrSe was shown to be 2.88 eV.

The Alkaline-earth Chalcogenides form a very interesting shell ionic system. These compounds have a crystalline rock salt structure (NaCl) at environmental temperature and pressure, see references. Application of pressure directly results in compression leading to the increased charge transfer (or three body interaction effect) [3, 4], due to the deformation of the overlapping electron shell of the adjacent ions (or non-rigidity of ions) in solids, this is as well known as induced pressure. A particular case of these compounds is Strontium selenide, which has been synthesized by molecular beam epitaxy, hot wall deposition, sputter deposition and floating melt zone. In the area of electroluminescent device doped SrSe thin films have been considered for the blue light source [5].

In this work a new methodology to synthesize strontium selenide on the base of reduction of selenium is presented. The selenium comes from oxidation stage 0 to -2, afterward it is mixed with a strontium salt (strontium chloride). In this process a fast substitution of chemical reaction is realized. The basic characterization of the material carried out by UV-vis, Raman spectroscopy, FTIR and transmission electron microscopy.

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2. Experimental

2.1 Chemical reaction synthesis

The fundamental reagents used to synthesize the nanostructured strontium selenide composite were:

- 1.- Aqueous solution of SrCl_2 (0.1M).
- 2.- A selenium ionic aqueous solution prepared by mixing 0.02 gr of elemental selenium powder with 2 ml deionized water plus 2 ml of concentrated (29%) of ammonium hydroxide and 2 ml of rongalite (1M).

This mix is heated in the range between 70-75°C until the selenium powder is completely dissolved.

The next step consists to combine the compounds prepared above described in same volumetric proportions. After the mixture is stirred in order to homogenize the chemical reaction, obtaining in this way the searched compound.

The last step is to centrifuge the compound in order to separate the solvents and after dry the sample at room temperature and then procedure with the characterization.

Optical absorption was measured with a Perkin Elmer UV-vis lambda 2 equipment.

The Raman dispersion measurement was carried out using a micro Raman X'plora BXT40 with a resolution of 2400T. The TEM used was JEOL JEM – 2010F. The FTIR spectrum was Nicolette, protégé-460.

3. Results

3.1 Optical absorption

In the figure 1 the optical absorption of SrSe thin film is shown. It is observed that the absorption edge is approximately of 250nm. The SrSe thin film exhibits relatively a low absorption. The left inset shows $[(\text{Abs})(\text{Energy})]^2$ vs ENERGY. The right inset shows $[(\text{Abs})(\text{Energy})]^{1/2}$ vs ENERGY. This lead to get the band gap values of 5.19 and 4.53 eV for direct and indirect transitions, respectively. These obtained values are close to some reported values calculated theoretically, [6].

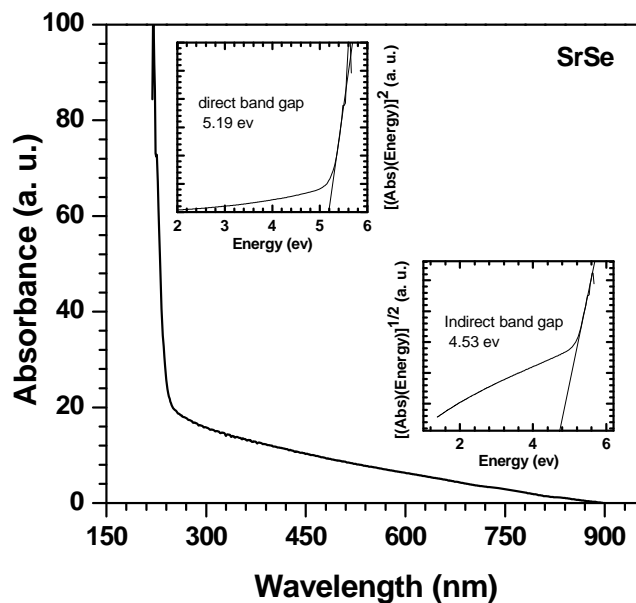


Fig. 1. Depicts an UV-Vis spectrum, corresponding to strontium selenide, the left inset corresponds to the direct energy bandgap giving a value of 5.19 eV and the right inset corresponds to the indirect energy bandgap with a value of 4.53 eV.

3.2 Raman spectroscopy

The Raman scattering is one of the main methods to obtain information on the kinds of phonon vibrations. The Raman spectra of strontium selenide in the 125-275 cm^{-1} region using a laser with 638 nm emissions, which is depicted in the figure 3. The higher peak located at 234 cm^{-1} corresponds to strontium selenide [6], the low signal at 114 cm^{-1} has been reported previously as confined mode for SrSe in [7]. The peak at 456 cm^{-1} corresponds to SrO [8],

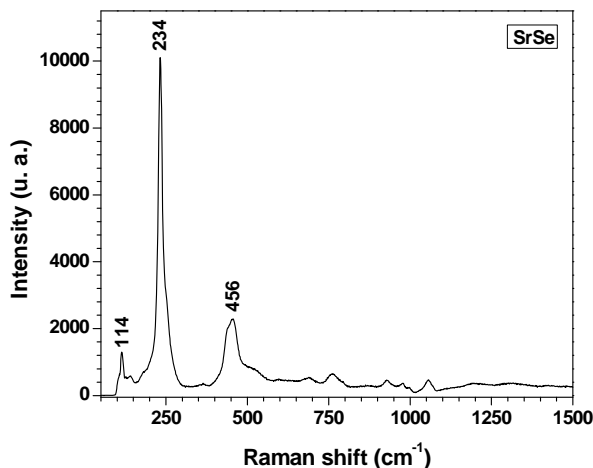


Fig. 2.- Dispersion Raman spectrum (638 nm), were the peaks 114 and 234 cm^{-1} have been identified as SrSe, while the signal at 456 cm^{-1} correspond to SrO.

3.3 Fourier Transform infrared

Fig. 3, corresponds to FTIR spectrum of the strontium selenide synthesized, here can be observed absorption frequencies at 3456 and 1610 cm^{-1} approximately, which correspond to the O-H vibration and bending vibration H-O-H respectively this is due to the absorption of H_2O in these samples. When these binary chalcogenides are exposed to the environment the water absorption is very common, so, this can to demonstrate that the compound is free of other solvents used to process the strontium selenide. The peak at 524 cm^{-1} is related to Sr^{2+} , and the formation of nanoparticles associated with the compound in question, this has been reported by Jenila et al. [9]. The absorption at 615 cm^{-1} was reported as strontium oxide [8].

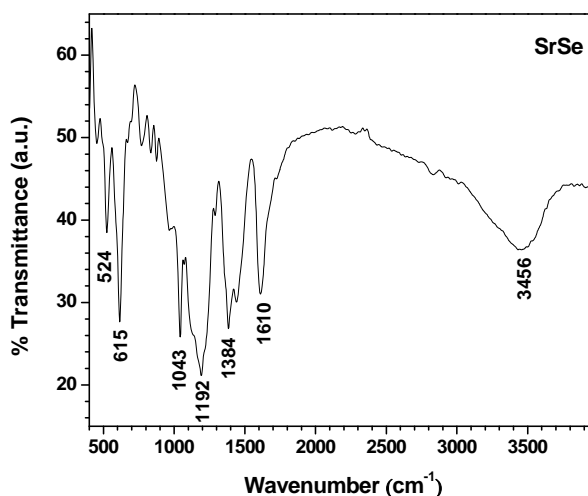


Fig. 3. FTIR spectrum for the obtained material SrSe, 1610 and 3456 cm^{-1} correspond to O-H and H-O-H vibrational modes. 524 and 615 cm^{-1} are Sr^{2+} and strontium oxide, respectively.

3.4 TEM images and its Fourier transform

Also TEM studies were realized to this material in order to identify the compound. Figure 4 shows two analysis TEM regions. First region corresponding to (a) and (b) is the image contrast and its Fourier Transformate, depicting low crystallinity, while the pair (c) and (d) correspond to a region of higher crystallinity and from this information was concluded that material match with cubic structure of strontium selenide JCPDS # 10-0182.

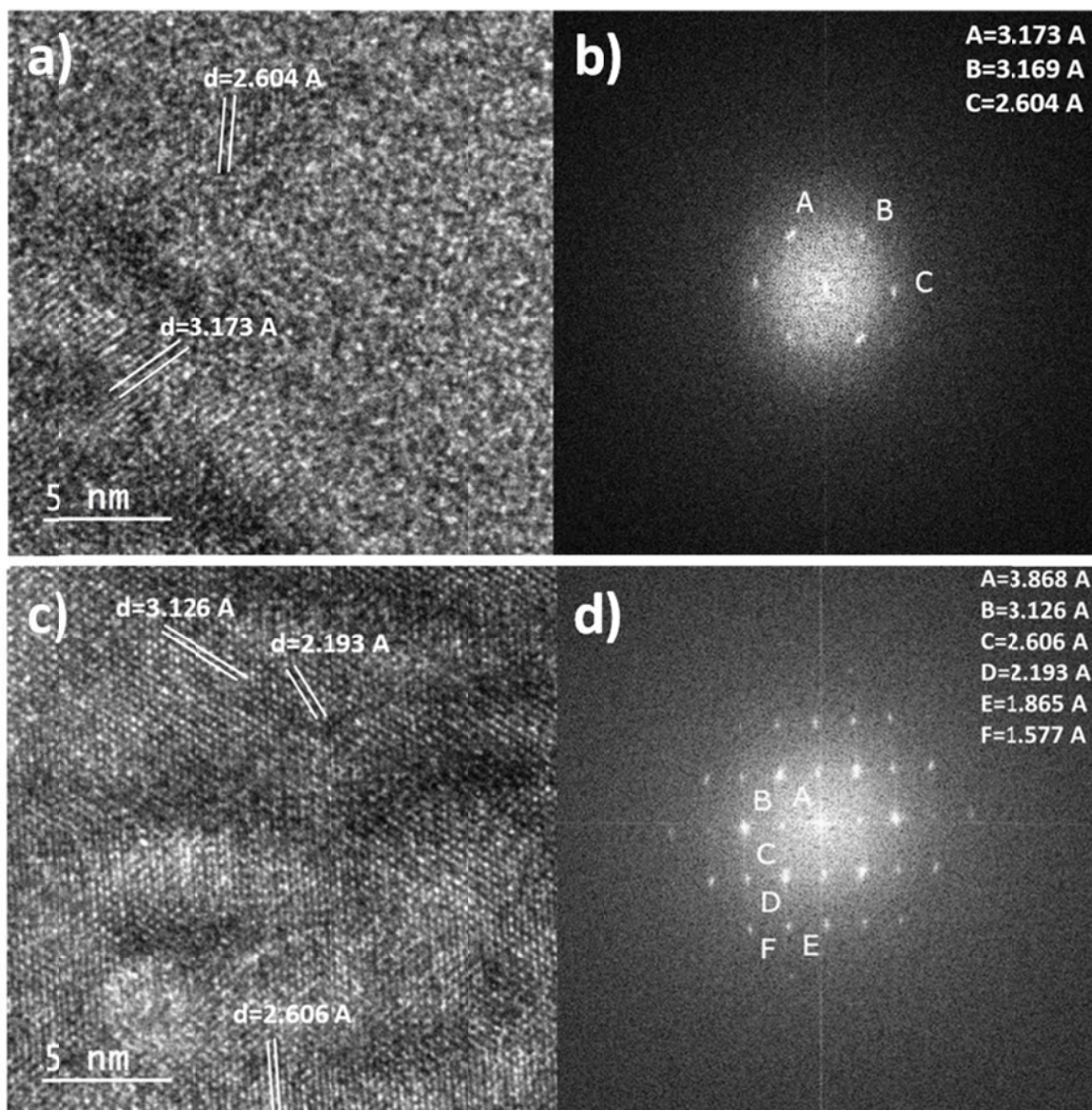


Fig. 4. Pair of micrographs with their Fourier Transforms, (a) and (b) represent one region on the material powder, while (c) and (d) were taken for other particle powder which shows higher crystallinity.

4 Conclusions

A procedure is described for the preparation of strontium selenide. In this work a novel process to synthesize SrSe was proposed. It seems to have a strong quantum confinement due to the direct band gap obtained, 5.19 eV. The well defined and main peaks were according to those reported in literature. The signals obtained by FTIR were partially identified for related compound with strontium element such as SrO, Sr(2+) and some nanoparticles formations.

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