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SYNTHESIS AND CHARACTERIZATION OF $SrTeO_3$ USING REDUCTED TELURIUM INTO A RONGALITE SOLUTION

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In this work a new methodology to synthesize strontium tellurite on the base of reduction of tellurium is presented. The Tellurium comes from oxidation stage 0 to -2, afterward it is mixed with a strontium salt, strontium chloride, in aqueous solution. In this process a fast chemical reaction of substitution it is realized. In order to make the optical characterization, the absorption in the UV-visible range was measured leading to compute direct and indirect band gap energies. The obtained strontium tellurite was a black powder after to be rinsed several times with deionized water, the obtained Band gaps values were 2.85 and 1.75 eV for direct and indirect respectively. From the Raman spectrum were identified three related peaks: at 118 cm^{-1} corresponding to SrO, 697 cm^{-1} corresponding to $(TeO_3)^{2-}$ (ν_3). and 776 cm^{-1} corresponding to $(TeO_3)^{2-}$ (ν_1). The infrared response shows SrO at 526 cm^{-1}, strontium oxide at 617 cm^{-1} plus O-H vibration and bending vibration H-O-H. Finally from transmission electron microscopy two crystallographic phases were detected, the monoclinic and the triclinic structures.

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1. Introduction

Some materials exhibit spontaneous electric polarization below the Curie temperature that can be reversed by the application of an electric field, these materials are called ferroelectric. Some applications of these materials are in electronics, photovoltaic effect, piezoelectric, in capacitors, lasers, in ionic superconductor and active biologic compound.

The Strontium Tellurite ($SrTeO_3$) is a ferroelectric material between 312 and 484 ^oC as is reported in [1]. Also, it has two important characteristics, one is that it can be used like a frequency duplicator and the other is that is the first compound with Tellurium, and it is used for phase equalization. Unfortunately, the method to obtain $SrTeO_3$ is very complicated with temperatures above 400 ^oC, and using controlled atmosphere with low pressure of vacuum. The Tellurium it is kept in an oxidation stage of -2, afterward it is mixed with a strontium salt (strontium chloride) where a fast chemical substitution reaction it is realized.

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The basic characterization of the material by UV-vis, Raman spectroscopy, the Fourier transform infrared spectrum (FTIR) and transmission electron microscopy (TEM) is carried out. In this work, a new simple method to obtain the $SrTeO_3$ is proposed, the synthesis is at room temperature and during a short period of time compared with reported literature [1-6].

2. Experimental

The fundamental reagents used to synthetize the nanostructured strontium Tellurite composite were: Aqueous solution of $SrCl_2$ (0.1M), a tellurium ionic aqueous solution prepared by mixing 0.02 gr of elemental tellurium powder with 2 ml deionized water plus 2 ml of sodium hydroxide (2M) and 2 ml of rongalite (1M).

This mix is heated stirring, in the range between 90-95°C until the tellurium powder is completely dissolved, and then the solution is kept in a closed bottle until to be used. The next step consists to combine the compounds prepared as above was described in same volumetric proportions. After they are aggregated, the mixture is stirred in order to homogenize the chemical reaction, obtaining in this way the searched compound. The last step consists to centrifuge the compound in order to separate the solvents then this was rinsed and centrifuged several times and dried the sample at room temperature to proceed with its characterization.

Optical absorption was measured with a Perkin Elmer UV-vis lambda 2 equipment, the Raman dispersion measurement was carried out by using a micro Raman X'plora BXT40 with a resolution of 2400T, TEM used was JEOL JEM – 2010F and the FTIR spectrum was measured by Nicolete, protégé-460.

3. Results

3.1 Optical absorption

In the Fig. 1 the optical absorption of $SrTeO_3$ nanostructured powder it is shown. There is observed that the absorption edge is approximately of 250nm. The $SrTeO_3$ exhibits relatively a low absorption. Typical experimental procedures and mathematical model were applied to obtain parameters from data in the selected range. The down inset shows $[(Abs)(Energy)]^2$ vs Energy. The up inset shows $[(Abs)(Energy)]^{1/2}$ vs Energy. This lead to get the band gap values of 2.85 and 1.75 eV for direct and indirect transitions, respectively.

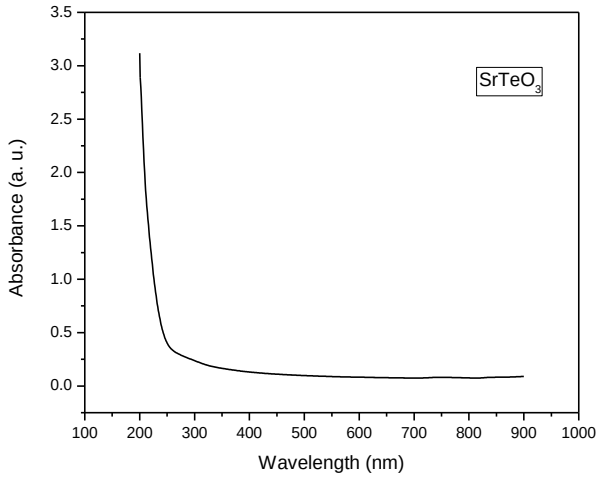

Fig. 1.- Depicts an UV-Vis spectrum, corresponding to strontium tellurite, the slight bending between 325 and 225 nm should correspond to electronic transition

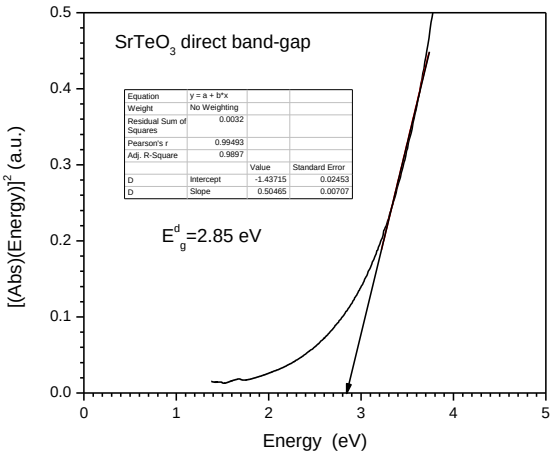

Fig. 2. Calculation of direct bandgap energy by mean of Tauc model-process from the experimental data.

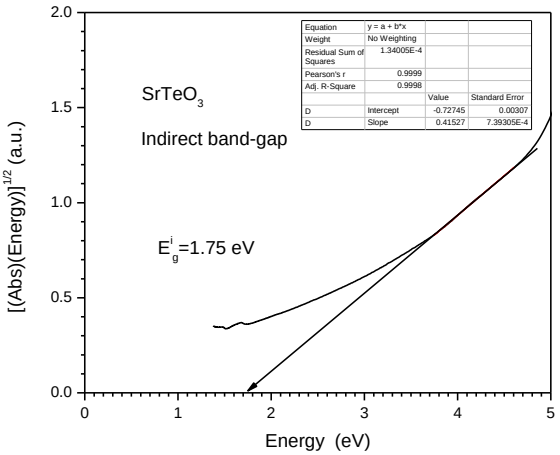

Fig. 3. Calculation of indirect bandgap energy by mean of Tauc model-process from the experimental data

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 tellurite in the 0-2000 cm^{-1} region using a 638 nm laser as a source is depicted in the figure 4. The higher peak located at 118 cm^{-1} corresponds to strontium oxide [7], the peak at 265 cm^{-1} could correspond with Raman dispersion of water [6], other related peaks are those in 402, 697 and 776 cm^{-1}, associated with TeO_3 vibrations [6, 8]. Other peaks shown in the same spectrum are summarized in the table 1.

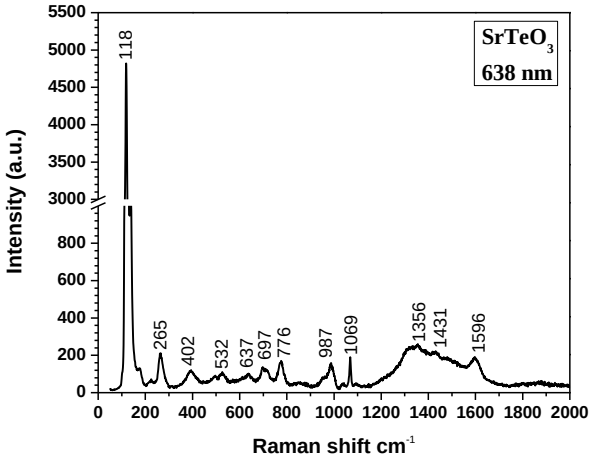

Fig. 4. Dispersion Raman spectrum (638 nm of Laser emission), from the powder of $SrTeO_3$ obtained

Table 1. Comparison of measured Raman shift versus reported values in bibliographic references.

Peak label	Raman shift (cm^{-1})	Reported Raman shift (cm^{-1})	Reference	Vibration modes
1	1069	1064	[9]	$\nu_3(SO_4)$
2	987	986	[9]	$\nu_1(SO_4)$
3	776	778	[9]	$\nu_1(TeO_3)$2-s
4	697	701	[9]	$\nu_1(TeO_3)$2-as
5	637	626	[10]	$\nu_4(SO_4)$
6	532	510	[9]	$\nu_2(SO_4)$
7	402	399w	[9]	δs (TeO_3) ($_2$)
8	265	270vw	[9]	$n_L(H2O_3)$
9	118	50-240	[9]	Sr-O

3.3 Fourier Transform infrared

Fig. 5, corresponds to FTIR spectrum of the strontium Tellurite synthetized, at this figure can be observed absorptions in the frequencies values of 3466 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 H2O in these samples[10]. When these perovskites-chalcogenides compounds 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 Tellurite. The peaks at 524 and 617 cm^{-1} are related to SrO [11, 12].

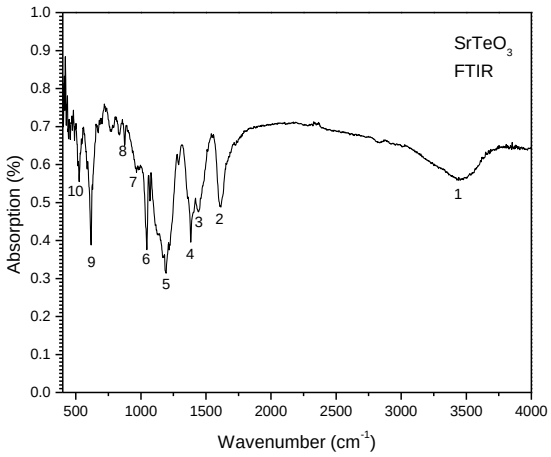

Fig. 5.FTIR spectrum for the obtained material $SrTeO_3$, 1610 and 3456 cm^{-1} correspond to water. 524 and 617 cm^{-1} are SrO signals

In table 2 are located the values of all labeled peaks of the figure 5, compared with values reported in references.

Table 2. Values of all labeled peaks

Peak label	wavenumber (cm^{-1})	Reported wavenumber (cm^{-1})	References	Assignments for
1	3466	3416	[11]	water
2	1614	1706	[12],[13]	ν(C-O), water
3	1441	1405	[14]	δ(C-H), C=Os δ(O-C-O)
4	1385	1401	[12]	NH4+ ιονσ
5	1194	1190	[9]	C-H and γ(C-H)
6	1047	1062	[14]	C-H and γ(C-H)
7	966	974	[9]	$\nu_1(SO_4)$
8	875	894	[14]	C-H and γ(C-H)
9	617	614	[11]	SrO
10	526	520	[10, 15]	SrO

3.4 TEM images and its Fourier transform

From the transmission electrons microscopy (TEM) technique we are capable to determine the morphology, the crystallographic planes and the Von Laue patterns, leading to know the structure of the synthetized compound, $SrTeO_3$. Also TEM studies help us to identify the chemical compound. Figure 6 shows two micrographs (a) and (b), which are contrast images showing morphologies of the poly-nanocrystals of $SrTeO_3$, at two different scales.

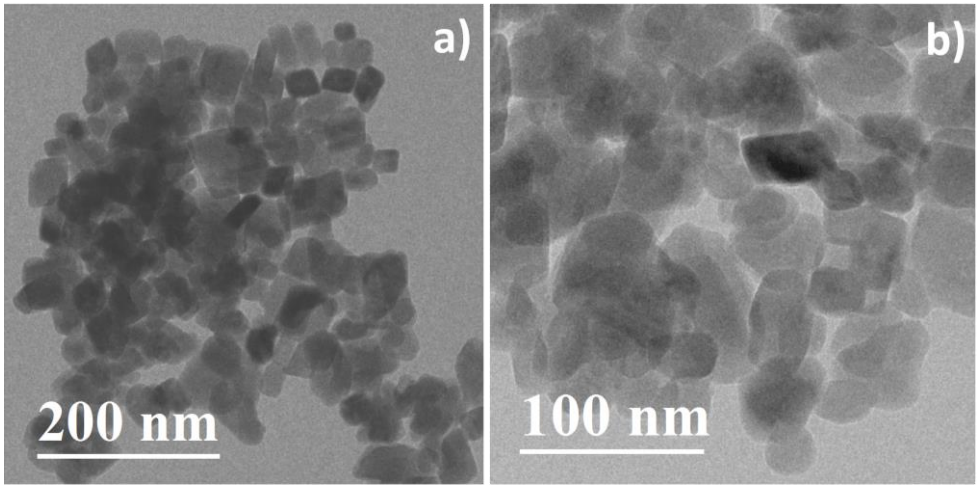

Fig 6. Shows panoramic views of contrast of the $SrTeO_3$ synthetized, a) is a region of the sample with less magnification, b) is a different region slightly higher magnified. Scales are indicated in each micrograph.

In figure 7 we are exposing both high resolution micrograph as well as its Fourier transform (corresponding to the of Von Laue pattern), see parts a) and b) of figure 7, respectively. In the part a), can be see a lot of crystallographic planes, and in part b) the pattern of its Fourier transform, which shows the two phases, monoclinic and triclinic of Strontium Tellurite ($SrTeO_3$). The monoclinic phase corresponds to the indices (202) and $(30\bar{2})$, while the triclinic phase is associated with the indices (002), (142) and (440). The Powder Diffraction Files (PDF) identified were PDF#48-1706 (Monoclinic) and PDF#35-1000 (Triclinic). The distances shown in part b) of Figure 7 were calculated using computer software for TEM.

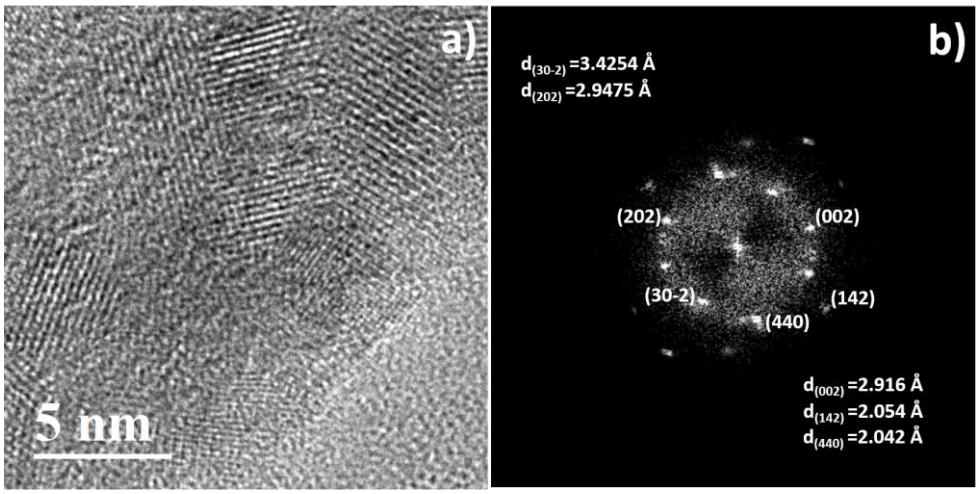

Fig. 7. Shown a micrograph with its Fourier Transform, (a) and (b), where it is possible to appreciate the crystallinity of the synthesized strontium tellurite

4 Conclusions

In this work a novel process to synthetize $SrTeO_3$ was proposed. The direct band gap obtained, 2.85 eV and indirect was 1.75 eV both in the visible region. The signals obtained by FTIR were partially identified for related compound such as SrO, TeO_3, SO_4, H_2O and some nanoparticles formations. The HRTEM lead identify mean reduced FFT two kinds of structures,

one monoclinic that correspond to PDF#48-1706 and a triclinic with PDF#35-1000. The easy form to synthetized the compound permit continue with the process if it is necessary doped to form more complicated compounds.

Acknowledgments

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