Scholars Journal of Applied Medical Sciences
Abbreviated Key Title: Sch J App Med Sci
ISSN 2347-954X (Print) | ISSN 2320-6691 (Online)
Journal homepage: https://saspublishers.com

OPEN ACCESS

Physics

Synthesis of Antimony Selenide (Sb_2Se_3) by Wet Milling: Structural Properties

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DOI: 10.36347/sjams.2022.v10i03.009 | **Received:** 14.02.2022 | **Accepted:** 17.03.2022 | **Published:** 22.03.2022

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Abstract **Original Research Article**

Single-phase bulk Sb_2Se_3 compound was synthesized by mechanical alloying using elemental precursors, Sb and Se. The collected sample has been successfully characterized by X-Ray diffraction for the wet mill to investigate the formation of Sb_2Se_3 phase. The XRD results analysis found that Sb_2Se_3 single phase was formed in 13 hours of milling. Raman shifts of 188 and 252cm^{-1} for wet as-milled Sb_2Se_3 had been recorded. EDS exhibits two strong peaks confirming the purity of our sample by detecting only Sb and Se elements, respectively.

Keywords: Ball milling, Sb_2Se_3, Wet milling, Synthesis, XRD.

INTRODUCTION

In a world with growing demand for energy, there is a need for identifying cost-effective and environmentally friendly solar absorber materials. Solar energy can play a tremendous role in meeting the required energy. Many solar cell materials like CIGS, CdTe, $CuSbSe_2$, Sb_2Se_3, $CuSbS_2$ etc., exhibit desirable characteristics. The solar cells of CdTe and CIGS have shown demerits in terms of the high cost of Indium and Gallium in CIGS and the toxicity of cadmium in CdTe, both ineluctable factors that shorten their commercial quantity of CdTe and CIGS absorber materials [1]. Antimony selenide (Sb_2Se_3) is a V–VI family of metals [1]. Having an orthorhombic crystal structure in which each Sb- and Se-atom is linked to three atoms of the opposite kind and held together in the crystal by weak secondary bonds. Sb_2Se_3 is a member of the space group Pnma 62. With a melting point of 5000 degrees Celsius, the elemental storage capacity of Sb and Se in the earth crust is 0.2 and 0.05 parts per million, respectively. Sb has a better storage capacity than competitors such as Indium (0.049 ppm) and Tellurium (0.005 ppm) [2]. It has thermoelectric properties and also serves as a pristine material for memory switching. Additionally, it is used to cover optical devices such as thermophotovoltaics, thermoelectrics, photodetectors, and topological insulators [3]. Antimony Selenide (Sb_2Se_3), a material that almost satisfies all the demand as a potential material for solar absorbers, has recently emerged [1]. It considerably attracted many researchers around the globe because of its low-cost, non-toxic, suitable and direct bandgap 1.22eV, earth-abundant constituents, high absorption coefficient (>10^5 cm^{-1}) at a wavelength slightly above the bandgap and excellent electronics properties, is a promising alternative absorber material for solar applications [4]. Furthermore, the Sb_2Se_3 compound is a layer structured semiconductor with an orthorhombic crystalline structure with covalent bonds formed by the reaction of Antimony with selenium. It has shown fair thermoelectricity power and good photo-conducting properties [5-6]. It has a unique one-dimensional ribbon structure [1, 6, 7]. A p-type semiconductor has hole mobility of up to 32 cm^2 V^{-1} S^{-1}, an electrical constant of 14.3 to 19.8 [1, 8]. Sb_2Se_3 is being synthesized via different routes starting from single-source precursor [9] hydrothermal reaction [10], physical vapour–liquid-solid (VLS) [7], solvothermal microwave-activated response [11], solid-state synthesis and ball milling [12].

We used mechanical alloying (wet milling) to improve batch homogeneity and consistency compared to other procedures. The sample may oxidize since the environment is not evacuated with argon or helium gas, and cold welding is less or not found due to the presence of solution.

The main purpose of this report is to provide information on the direct synthesis of single-phase bulk antimony selenide (Sb_2Se_3) by mechanical alloying (wet milling).

EXPERIMENTAL DETAILS

Elemental powders of Antimony (99.99%) and selenide (99.99%) were purchased from SRL. The stoichiometry ratio of 2:3 was prepared and treated in a planetary ball mill (Pulverisette 6, Fritsch; rotational speed 500 rpm; tungsten carbide bowls of volume 250 ml; 25 number tungsten carbide milling balls of diameter 10 mm; under argon atmosphere). A process control agent (Toluene) was added to the contents of the jar. The whole process lasted for 13 hours, with the first sample being collected after the first 5 hours. After that, the sample was collected after every 120 minutes. The Sb_2Se_3 phase formation was detected by high-resolution X-ray diffraction (X-pert pro diffractometer) operated at 40 kV and 30 mA, Raman scattering spectroscope (Reinshaw invia Raman Microscope) with an Olympus microscope equipped with a 100X magnification lens and in the backscattering configuration. The excitation source was a green Argon ion laser operating at 532 nm and 220 mW output powers. Field Emission Scanning Electron Microscope inspected the morphology of the films (FET Quanta USA) operated at 30 kV at a magnification of 10,000X.

RESULTS AND DISCUSSION

Figure 1 shows a typical XRD pattern of as-milled (wet) Sb_2Se_3. After 5 hours of milling, Sb_2Se_3 started forming with Sb, the main phase with elemental Se. With the increase in milling time of 2 hours, Sb_2Se_3 peak start becoming intense while the Se peak diminished. With the increase in milling time, more energy is supplied to the Sb-Se powder to form Sb_2Se_3. The increase in the milling time from 7 to 9 hours, Sb remains the main phase. The same pattern has been observed with additional milling time of 2 hours. At 13 hours of milling the single phase Sb_2Se_3 (2 1 2) appeared at $2\theta = 31.25136^0$. The FWHM and the crystallite size were determined at 0.39325 and 210.13 respectively. The different peaks existing shown the nature of bulk Sb_2Se_3 single phase. The Sb_2Se_3 can be compared with the standard diffraction pattern for Sb_2Se_3 (JCPDS N0: 04-006-2232).

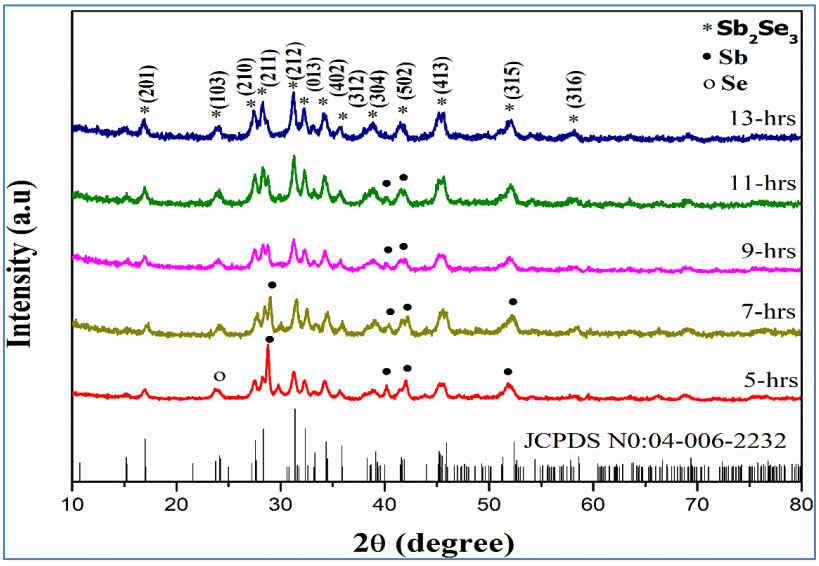

Fig-1: XRD pattern for wet-milled samples

In order to confirm the single-phase formation obtained from XRD, Raman spectroscopy was conducted, as shown in Figure 2. The Figure indicates a pure Sb_2Se_3 phase exhibiting two strong peaks, Raman shifts of 188 and 252cm^{-1} for wet as-milled Sb_2Se_3 (13 hours of milling)

The Raman shifts of 188 correspond to Se-Se bond and 252cm^{-1} correspond to Sb-Sb bond respectively. These Raman bands are characteristics of Sb_2Se_3 previously reported [13, 14].

The crystallite size was calculated using equation (1) from [15]

$$\boldsymbol{D = \frac{0.9\times\lambda}{\beta\times\cos\theta}} \quad \textbf{..... (1)}$$

Where D is the crystallite size, λ is the radiation wavelength, β is the FWHM in radians and θ is the Bragg's angle.

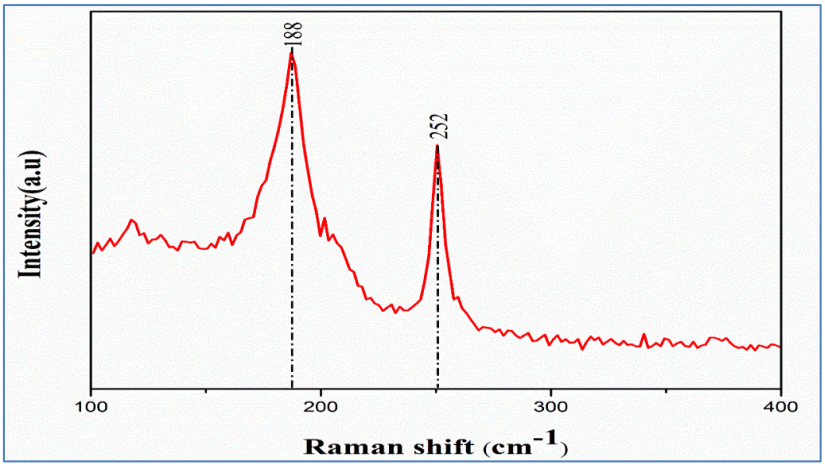

Fig-2: Raman Spectra of as-milled (wet) Sb_2Se_3

The surface of the sample wet milled for 13 hours is shown in Figure 3. The surface appeared cloudy with large grains.

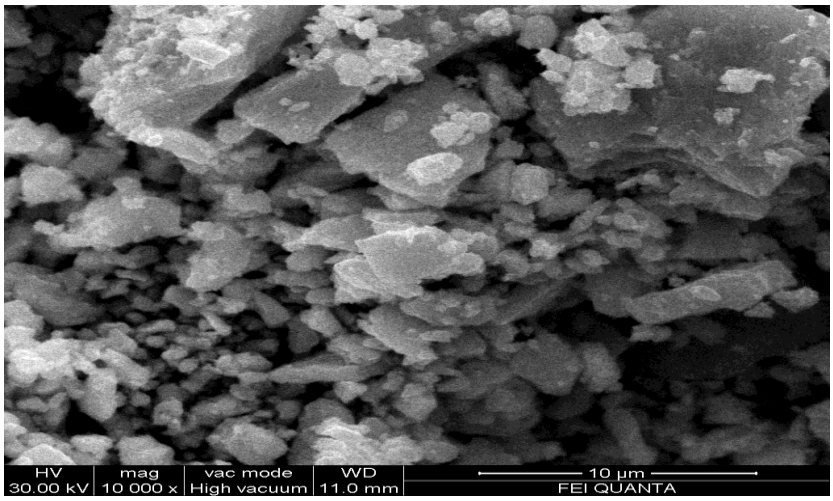

Fig-3: FE-SEM micrographs of the wet milled (13 hours) sample

In order to complete the FESEM observation and measurement, Energy dispersive x-ray spectrum (EDS) was conducted on the sample as shown in Figure 4. The Figure exhibits two strong peaks confirming the purity of the sample by detecting only Sb and Se elements respectively. From the EDS image, it is confirmed the atomic ratios of elemental Se are close to 2:3 as expected from stoichiometry ratio.

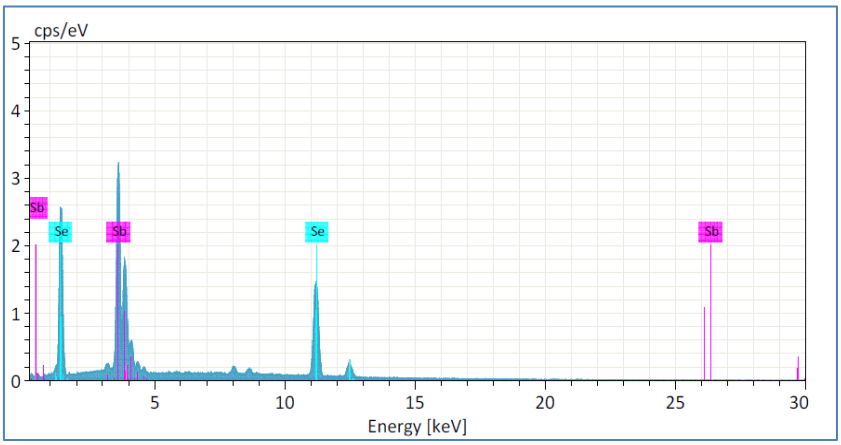

Fig-4: EDS of wet as-milled Sb_2Se_3

Table-1: Elemental composition of the wet milled sample

Element	Mass%	Atomic%	Abs. error (%)	Rel. error (%)
Sb	55.41	44.62	1.53	3.00
Se	44.59	55.38	1.06	2.58
Sum	**100.00**	**100.00**	**2.75**	**5.58**

CONCLUSION

Mechanical alloying is a technique involving repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill and thus all the structural and chemical changes are produced by mechanical energy. The as-milled powders of Sb_2Se_3 were studied using various tools such as XRD and Raman spectroscopy to understand the structural properties of wet as-milled synthesized Sb_2Se_3. It has been found out that the single phase of Sb_2Se_3 can only be realized after 13 hours of ball milling.

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