



7-23-2021

## Advancement in Infrared Optics Through the Exploration of Solution Derived Arsenic Selenide (As<sub>2</sub>Se<sub>3</sub>) Thin Films

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### Recommended Citation

Orsini, Annabella, "Advancement in Infrared Optics Through the Exploration of Solution Derived Arsenic Selenide (As<sub>2</sub>Se<sub>3</sub>) Thin Films" (2021). *Physics and Astronomy Summer Fellows*. 35.  
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# **Advancement in Infrared Optics through the Exploration of Solution Derived Arsenic Selenide (As<sub>2</sub>Se<sub>3</sub>) Thin Films**

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July 2021

## **Abstract**

There are great opportunities for advancement in the realm of infrared (IR) optics through the use chalcogenide glasses (ChGs). The development of IR optics using ChGs is important for applications in search and rescue operations, firefighting efforts, medical imaging, and satellites. Instead of creating bulky, expensive, single crystal IR glasses, ChGs can be deposited as thin films by solution derived (SD) spin or dip coating. Our research takes a multidisciplinary approach to investigate ChGs thin films using physics, chemistry, optics, and materials science. Specifically, we use arsenic selenide ( $\text{As}_2\text{Se}_3$ ) dissolved in ethanolamine to create thin films through both the spin-coating and dip-coating processes. These films are then tested for stability and IR transmission capabilities. The dissolution of  $\text{As}_2\text{Se}_3$  in ethanolamine and the creation of thin films is a process that has not been as deeply explored in comparison to other solution derived ChG materials. This research will provide framework for the future use of  $\text{As}_2\text{Se}_3$  based solutions when creating IR thin films.

## **I. Introduction**

Today, glass has emerged as a high-technology material with applications in fiber optics for high-speed communication, in medical treatments as bone-setting materials, and in optical devices such as precision lenses<sup>1</sup>. Our research will focus specifically on chalcogenide glasses, also known as ChGs. Chalcogenides are created using Group 14 elements such as sulfur, selenide, and telluride. Tellurides are often used in current chalcogenide technologies. However, this material can be expensive as well as less flexible than sulfide and selenide compounds. These elements are dissolved in amine-based solvents. Previous research on solution processed ChG films uses  $As_2S_3$  or less often  $As_2Se_3$  material dissolved in ethylenediamine<sup>2-4</sup>. We propose the use of ethanolamine to dissolve  $As_2Se_3$  and create thin films. In the current literature, there is a significant lack of this specific method of creating chalcogenide films. Our research, we predict, will produce viscous solutions that will then in turn allow to thicker films that will have greater forms of application. Ethanolamine was found to dissolve compositions containing As, S, Ge, and Sb<sup>3</sup>. The table below represents the properties of ethanolamine in comparison to other solvents. Additionally, due to the high volatility of ethylenediamine and propylamine only ethanolamine solutions were suitable for Raman spectroscopy measurements<sup>3</sup>.

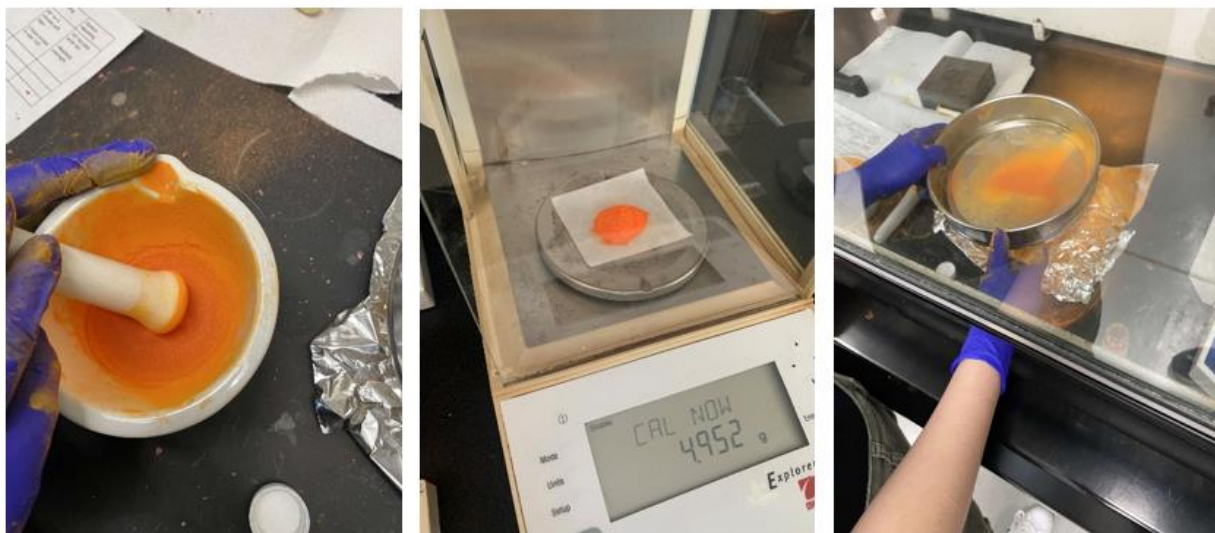
**Table I.** Summary of amine solvent properties<sup>3</sup>.

Solvent	<b>Propylamine</b> H <sub>2</sub> N-CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>3</sub>	<b>Ethylenediamine</b> H <sub>2</sub> N-CH <sub>2</sub> -CH <sub>2</sub> -NH <sub>2</sub>	<b>Ethanolamine</b> H <sub>2</sub> N-CH <sub>2</sub> -CH <sub>2</sub> -OH
M.W. (g/mol)	59.11	60.103	61.08
Density (g/cm <sup>3</sup> )	0.719	0.899	1.012
Boiling Point (°C)	48	116	170
Basicity (pK <sub>b</sub> )	3.4	4.1	4.5
Viscosity (cP)	0.359	1.54	19.4

## **II. Experimental Procedure**

### **Arsenic Sulfide (As<sub>2</sub>S<sub>3</sub>)**

Before primarily using As<sub>2</sub>Se<sub>3</sub>, the first attempt was to create films using As<sub>2</sub>S<sub>3</sub>. Unfortunately, issues arose with the powdering yield during the solution making process. Initially, we had a bulk sample of about 25g. This large bulk piece of As<sub>2</sub>S<sub>3</sub> was then broken into smaller pieces carefully with a hammer. Using the mortar and pestle, the bulk As<sub>2</sub>S<sub>3</sub> pieces were ground into a fine powder. The powdering process was completed under the protection of the fume hood, so the glass did not become a hazard. Once a fine orange powder was achieved, it was then transferred to a vial to be weighed on the scale before sifting. The powder was then sifted through a 125-micron sieve onto tinfoil. After sifting, what yield available was transferred to a vial. From about 5g of not sifted powder, we were only able to yield about 3g after the sifting process. This was not enough yield to create our solutions with ethanolamine. It was at this point that it was recognized there were external factors within the sifting process that was causing a significant loss of As<sub>2</sub>S<sub>3</sub>. The decision was then made to switch our methods and begin using arsenic selenide instead.



**A**

**B**

**C**

**Figure 1:** **A)** Powdering of bulk  $\text{As}_2\text{S}_3$  in the fume hood using the mortar pestle. **B)** Weight of the yield after powdering and before the sifting process. **C)** Sifting the powdered  $\text{As}_2\text{S}_3$  using the sieve.

### **Arsenic Selenide ( $\text{As}_2\text{Se}_3$ )**

Since there were issues with the sifting process when we used  $\text{As}_2\text{S}_3$ , we made the decision to negate that part of the procedure for the use of  $\text{As}_2\text{Se}_3$ . First, the same powdering process that was used with the arsenic sulfide was used for the selenide compound. In this case, the initial bulk sample of  $\text{As}_2\text{Se}_3$  weighed 17g. Again, the bulk glass was ground up in the fume hood using the mortar and pestle. Because the sifting process was negated, we were careful to spend extra time grinding down the bulk sample to eliminate the possibility of larger pieces remaining in the powder that could affect our results. Once powdered, the  $\text{As}_2\text{Se}_3$  was transferred to a weigh boat to measure the yield on the scale. A high yield was achieved from this process at 16.752g of powder from the 17g bulk glass.



**A**

**B**

**C**

**Figure 2:** **A)** Grinding the  $\text{As}_2\text{Se}_3$  in the mortar and pestle. **B)** Vial with not sifted powdered  $\text{As}_2\text{Se}_3$ . **C)** Final yield from powdering the bulk sample.

Post-powdering, it was time to create the solutions. Approximately 6.5g of the  $\text{As}_2\text{Se}_3$  powder was used and split amongst four vials. Below is a table that reflects the amount of powdered arsenic selenide and liquid ethanolamine used for each solution.

**Table II. Solution Information**

Solution Number	Amount of $\text{As}_2\text{Se}_3$ (g)	Amount of ethanolamine (mL)	Revised amount of ethanolamine (mL)	Concentration (g/mL)
1	0.504	5.0	15.0	0.033
2	1.030	5.0	15.0	0.066
3	2.042	5.0	20.0	0.10
4	2.999	5.0	X	X

Once the initial solutions were created, each with 5mL of ethanolamine, they were spun on the hot plate at room temperature using the magnetic stirrer for 24 hours. The

four vials were stabilized on the hot plate using a clamp, which was all placed under the fume hood for safety.



**A**

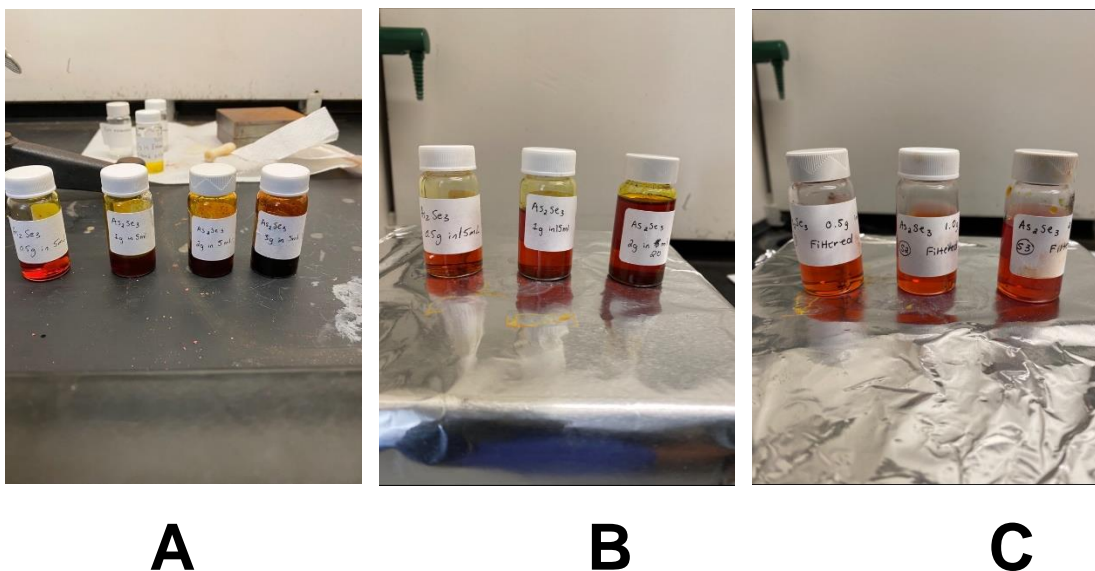


**B**

**Figure 3: A)** The four solutions created before stirring. **B)** The solutions secure and stirring on the magnetic plate.

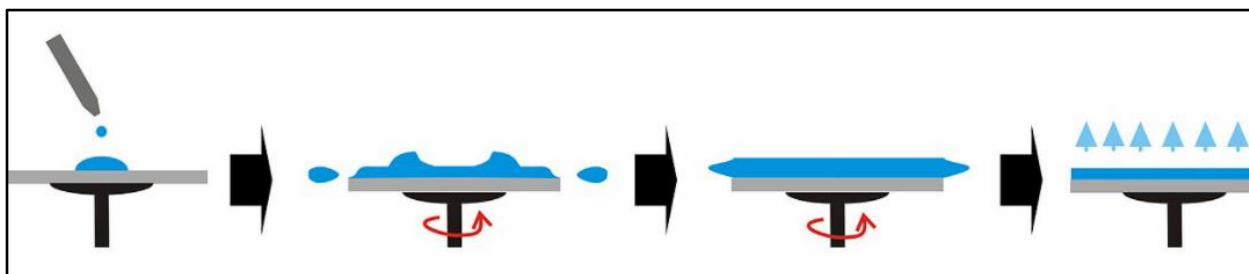
The viscosity and clarity of the solutions were observed once they were done stirring. It was discovered that a significant amount of precipitate remained at the bottom of all four vials. To appease this, additional ethanolamine was added to the solution. The amount added to each solution is reflected in Table II. It was also at this point that it was decided to disregard the fourth solution due to the excessive amount of precipitate that remained. The solutions were again left to stir on the magnetic plate for an additional 24 hours. Afterwards, a significant decrease in the amount of excess precipitate in the vials was observed. The little amount of remaining precipitate was then filtered out using a 0.5-micron filter. Each solution was carefully filtered into a fresh vial. The filtering process yielded nicely colored viscous solutions with no observed precipitate.





**Figure 4. A)** The four solutions after the initial spin with 5mL of ethanolamine. **B)** The remaining three solutions after revisiting with more ethanolamine. **C)** The solutions post-filtering.

Now that we had our three well-made solutions (Fig. 4), the dip and spin coating processes began. For dip-coating, small pieces of substrate were coated in each solution for 24 hours, as well as one large substrate in the second solution for a short period (Table III). Large slide substrates were then placed in the spin-coating machine one by one (Fig. 5). Each solution had two corresponding substrates that were spun at 2000 rpm and 3000 rpm, respectively, for a total of six spin-coated samples (Table IV).



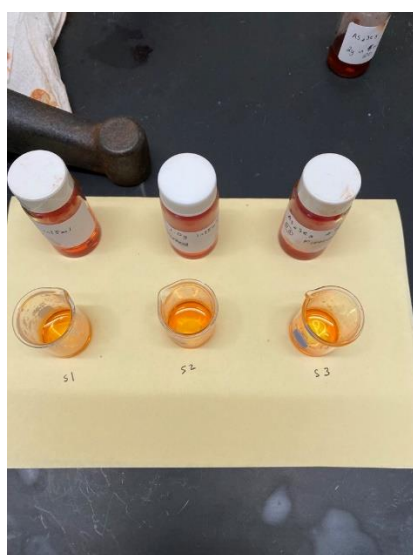
**Figure 5.** Spin-coating procedure diagram<sup>5</sup>.

**Table III.** Dip coated sample concentration.

Sample	Con. (g/mL)	Dip Time (hrs)
D1	0.033	24
D2	0.066	24
D3	0.10	24
D4	0.066	1

**Table IV.** Spin-coated sample concentrations.

Sample	Con. (g/mL)	RPM
S1-2	0.033	2000
S2-2	0.066	2000
S3-2	0.10	2000
S1-3	0.033	3000
S2-3	0.066	3000
S3-3	0.10	3000



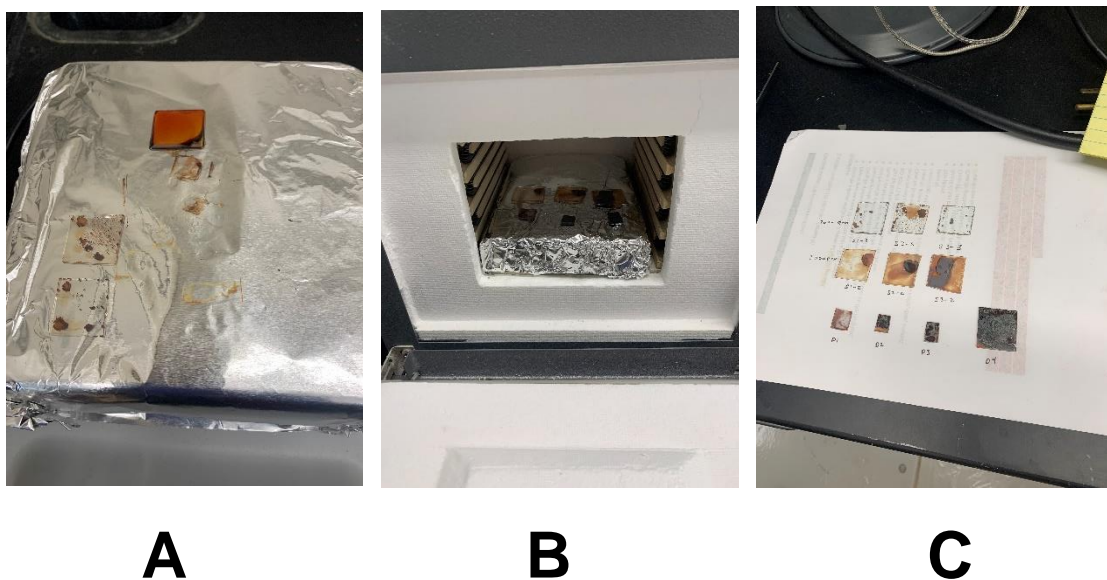
**A**



**B**

**Figure 6.** **A)** Dip-coating the substrates in each of the three solutions, covered to avoid evaporation. **B)** A coated substrate placed in the spin-coating machine.

Once the substrates were fully coated, they were all then moved to the hot plate to begin the baking process. This began with a soft bake at 90°C on the hot plate for five minutes. The larger dip-coated substrate was soft-baked for one hour. All samples were then carefully transferred to the oven using tweezers to bake at 120°C for two hours. After fully baking, the coated substrates were taken out and cooled before being tested for transmission capability.



**Figure 7.** **A)** Substrates soft baking on the hot plate. **B)** Substrates hard baking in the oven. **C)** The resulted films from the baking process.

The films in the second row of image C in Figure 7, which are the films spun at 2000 rpm, as well as the large dip-coated substrate in the bottom right of the image were the films we used in our transmission analysis. The four films were placed into the spectrometer individually. For this task, a FTIR, or Fourier transform infrared, was used as a spectrometry tool. To create a background for the transmission data, a blank slide of borosilicate glass was used. The transmission data was taken at a sample size of 0.5cm, and measured between 1500 and 5000 nanometers into the IR. For each

substrate, the percent transmission for both a light and dark region of the film was measured. For each measure collected, the individual transmission data was added into one large graph to see how their transmission capabilities compared.

### **III. Results and Discussion**

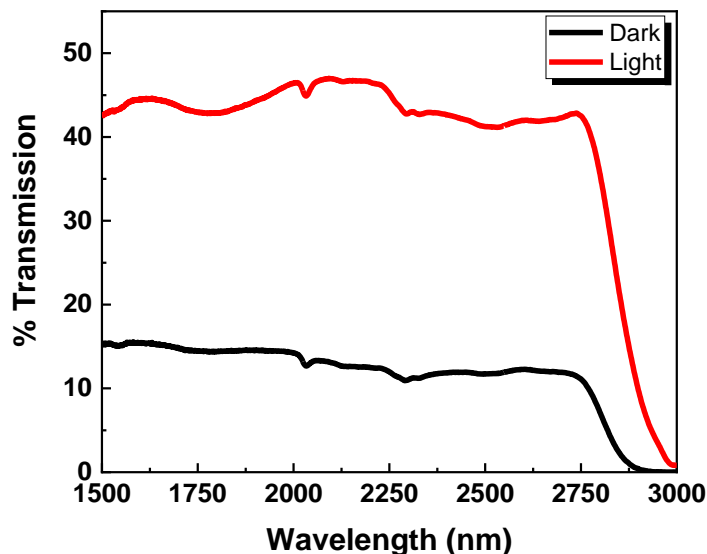
The film creation process resulted in ten total films, four dip-coated and six spin-coated. After observing the quality of the films after baking, it was clear that the films made from spin-coating at 3000 rpm were not going to produce good transmission capabilities. This was also true for the smaller dip-coated samples.

Unfortunately, the films did not end up as homogeneous as was predicted, and they had delaminated during the baking process. There are some factors that we believe could have contributed to the delamination. Firstly, there could have been an issue with the concentrations of our solutions. Since there was little to no literature on this specific combination of  $As_2Se_3$  and ethanolamine, our solution recipes could not be fine-tuned yet. Also, the spin-coating process could have been done at too high of a speed, and therefore the thin layer on the substrate did not have the ability to fully attach to it. There could also have been issues with the substrate itself, as the substrate was not prepped in any way before creating the films.

#### **Dip-coated Sample Results**

The transmission data from the dip-coated sample, created from the second solution, revealed a reduction in transmission for both the light and dark regions measured. The dark region of the sample produced about fifteen percent transmission into the IR and the light region hovered between forty and forty-five percent transmission.

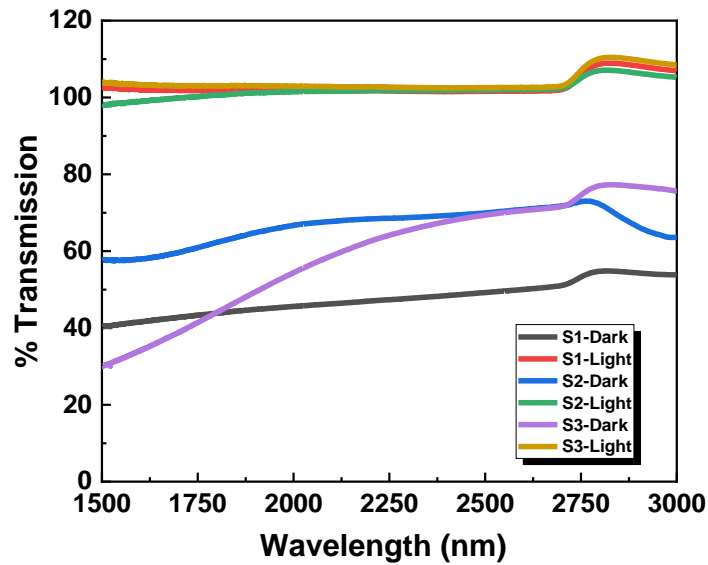
These results are not unexpected, as this process is new territory regarding the use of ethanolamine in combination with  $\text{As}_2\text{Se}_3$ .



**Figure 8.** Transmission data for D4 dip-coated sample.

### Spin-coated Samples Results

For the spin-coated samples, three films were tested for their transmission abilities. Since the films were not homogeneous on the substrate, light and dark regions were measured separately. The samples spun at 2000 rpm are labeled S1-2, S2-2, and S3-2. Those spun at 3000 rpm are labeled similarly with a '-3'. For all three of the films, the light regions were measured to have a percent transmission nearing one hundred. The dark regions of all the films showed a reduction in transmission. For the dark region of S3-2, the one created from the third solution, there was some evidence of scattering. This was likely due to environmental factors, such as air pockets or issues with the substrate.



**Figure 9.** Transmission data for spin-coated samples.

#### **IV. Conclusion**

Although our work did not produce homogeneous thin films as we had hoped, we were successful in creating a procedure for creating nice viscous solutions using and  $As_2Se_3$  ethanolamine. This procedure will be significant for any future work using these particular solvents and solutes. In the future, we hope to acquire a more successful baking and film creation process so that our thin films are homogeneous and stable in their transmission capabilities. There is also work needed to create a process that will improve substrate adhesion.

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