Synthesis of Novel Bi / Sb Modified Chalcogenide Glasses

Travis Hodge , Roman Golovchak

Austin Peay State University, Department of Physics and Astronomy, Clarksville, TN, USA

Abstract

Chalcogenide glasses (ChG) are considered as the most convenient and inexpensive media for applications in modern photonics, combining high IR transparency, excellent fiber drawing capability and largest optical nonlinearities reported to date. Three different systems of these chalcogenide glasses were prepared to determine the ability to form an amorphous sample. The systems consisted of : $Bi_x(GeSe_4)_{33-\frac{x}{3}}(GeTe_4)_{33-\frac{x}{3}}(GeS_4)_{33-\frac{x}{3}}$ (x = 0,1,5), $Bi_x(GeSe_4)_{60-\frac{x}{3}}(GeTe_4)_{20-\frac{x}{3}}(GeS_4)_{20-\frac{x}{3}}$ (x = 0,1,5) , and $Bi_xGe_{20}Sb_{20-x}Se_{20}Te_{20}$ (x = 0,1,5). These sample were prepared by the conventional melt-quench method with high purity (5N) precursors. Nine Samples were synthesized resulting with two amorphous samples in the $Bi_x(GeSe_4)_{60-\frac{x}{3}}(GeTe_4)_{20-\frac{x}{3}}(GeS_4)_{20-\frac{x}{3}}$ (x = 1,5) system. These results were then confirmed with both X-ray Diffraction (XRD) and thermal analysis.

Glass Systems

The synthesis process began by splitting the multi-chalcogen glasses into three distinct systems. For each system, we would like to produce three samples, when x = 0,1,5. These three systems were :

- $Bi_x\,(GeSe_4)_{33-\frac{x}{3}}\,(GeTe_4)_{33-\frac{x}{3}}\,(GeS_4)_{33-\frac{x}{3}}$
- $Bi_x\,(GeSe_4)_{60-\frac{x}{3}}\,(GeTe_4)_{20-\frac{x}{3}}\,(GeS_4)_{20-\frac{x}{3}}$
- $Bi_x\,Ge_{20}\,Sb_{20-x}\,Se_{20}\,Te_{20}$

These systems were chosen for testing the glass forming abilities in new compositions. For these systems, the synthesis process is still experiment; as a result, different methods of producing these glasses would also be tested in an attempt to optimize the final sample in each system.

Distillation of Sulfur

In order to maintain the purity of each sample in the produced systems, we needed to purify the sulfur used. Starting with 180 grams of sulfur, we distilled the solid sulfur and condensed the pure sulfur product. This process was completed using an arrangement of three different heating systems to properly melt and boil the sulfur as well as condense it back into a purified sulfur product. The produced sulfur is shown in figure 1.

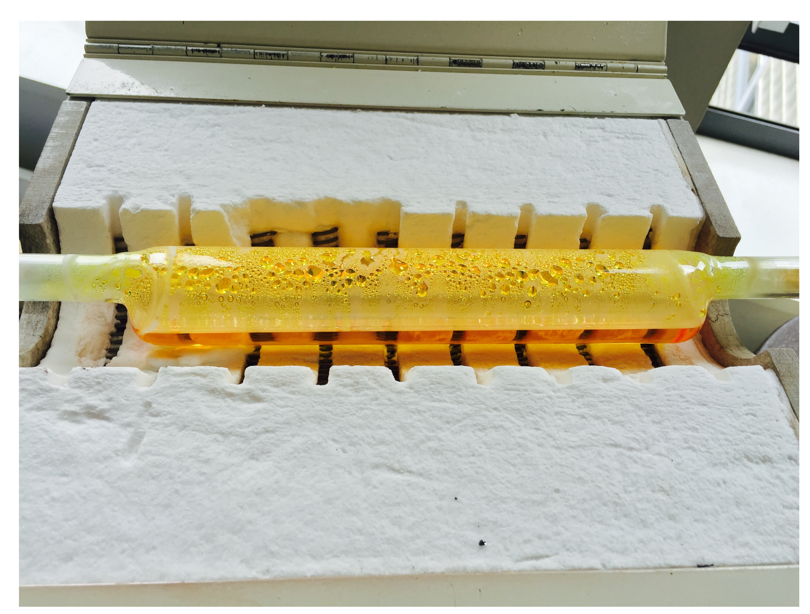

Figure 1: The produced purified vessel of liquid sulfur. This sulfur was then solidified as used in the production of the sulfur based ChG.

Synthesis of Glass

In order to produce these systems, all production materials (sample tubes, tools, instruments, etc.) must be cleaned. In cleaning the sample tubes, HF acid was used to remove impurities from the tubing. After the initial cleaning, remaining water and oxygen were removed by vacuum (10^{-6} mbar). The materials and sample tubes were then placed in a glove box for transfer. After the sample tubes were filled with the correct stoichiometric ratios, the tubes could then be placed in a rocking furnace for heating. According the to sample reference, the furnace then followed a programmed heating method.

Synthesis Cont.

Due to the unknown glass transition temperature of the glass systems, various heating methods were used in an attempt to produce an amorphous solid.

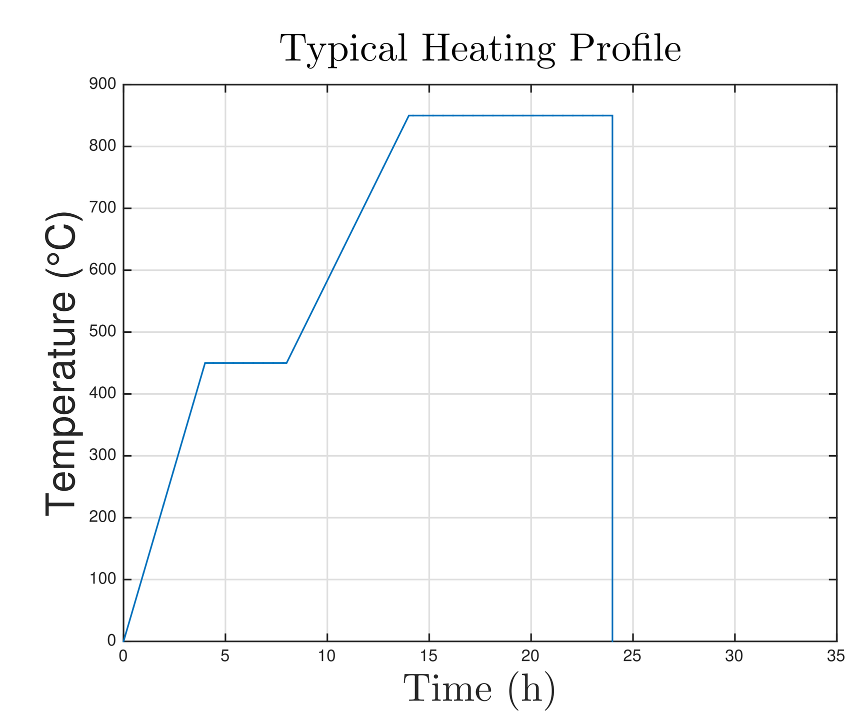

Figure 2: Typical thermal profile used in synthesizing ChG.

Table 1: Sample References for Each System

Ref.	Bi_x	Composition
H_1	1	$Bi_1Ge_{19.8}Se_{26.4}Te_{26.4}S_{26.4}$
HB_0	0	$Ge_{20}Sb_{20}Se_{20}Te_{20}S_{20}$
HC_5	5	$Bi_5\ Ge_{18.9}Se_{46.6}Te_{14.6}S_{14.6}$

Results

Figure 3: The final amorphous samples produced (HC_1 & HC_5).

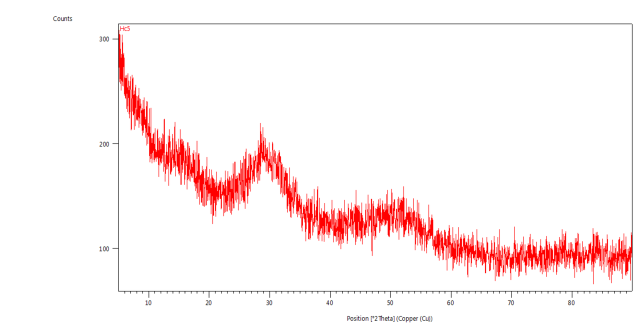

Figure 4: Shows the wide peak width, constituting an amorphous solid.

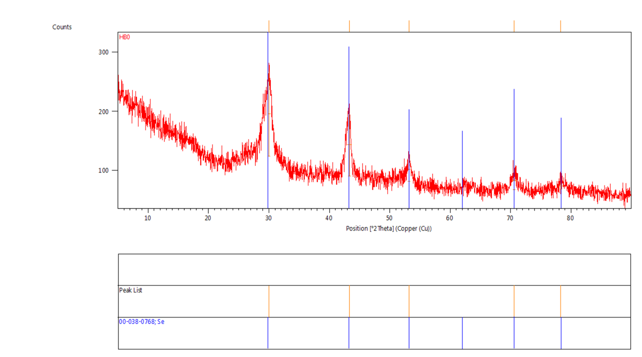

Figure 5: Shows the sharp peaks associated with Se crystals.

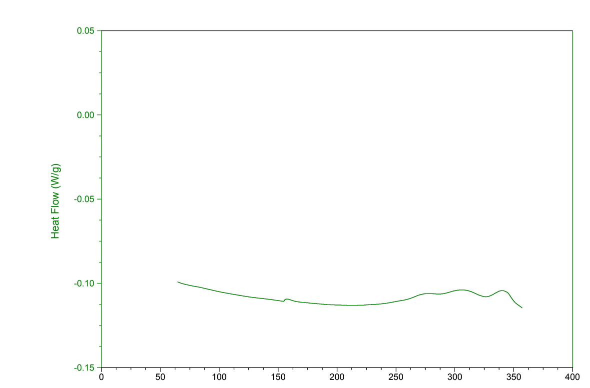

Figure 6: Shows no visible glass transition temperature

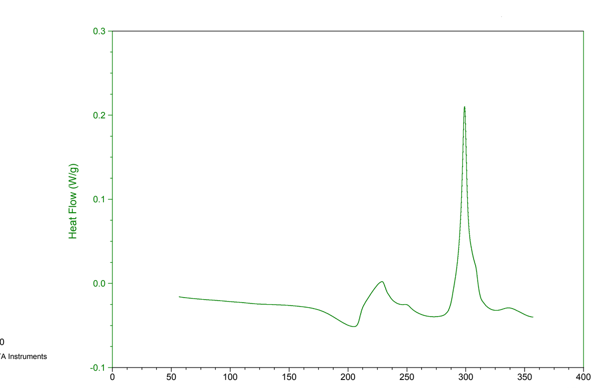

Figure 7: Shows a glass transition temperature at 177°C.

Conclusion

After adjusting the thermal profile and stoichiometric compositions, we produced an amorphous solid in the BGTSeS system. It was determined that a smaller sample tube diameter and quenching horizontally aided in the production of a glass.

Acknowledgements

- The authors would like to acknowledge the U.S. National Science Foundation through the International Materials Institute for New Functionality in Glass at Lehigh University, grant DMR-0844014.
- The authors acknowledge the Glass and Ceramics group (Bruno Bureau and Catherine Boussard-Pledel) at Rennes for providing the lab equipment and materials for the research.