SPS Chapter Research Award Interim Report

Project Title	Ionic Conductivity of the Lithium Clustering Effect
Name of School	Coe College
SPS Chapter Number	1255
Total Amount Awarded	\$2,000.00
Project Leader	Anne Ruckman

<u>Abstract</u>

The Coe College SPS Chapter will investigate the lithium clustering effect on silicate and borate glasses for better understanding the ionic diffusion due to electric fields and ionic conductivity. SPS members will learn how to prepare glass samples, conduct electrical impedance spectroscopy measurements, and witness the novel lithium clustering effect.

Statement of Activity

Interim Assessment

Research Question

The objective of this research is to better understand the clustering effect of lithium in lithium borate glasses and its effects on ionic conductivity. We are especially interested in compositions containing high concentrations of lithium. This research will expand our understanding of glass properties, expose us to novel conductivity testing methods and analysis, as well as strengthen our relationship with the Glass and Optical Research Group, part of the Materials Science and Engineering Department, at Iowa State University (ISU). We will study chemical glass formations and gain hands-on research experience with novel testing equipment in materials science development. This includes glass characterization, such as density measurements, differential calorimetry scanning, impedance spectroscopy at low frequencies (1-100,000 Hz), and X-ray diffraction.

Brief Description of Project

The Coe College SPS Chapter aims to address the ion conductivity plateaus with lithium borate systems with high lithium content. We paired experienced SPS researchers with introductory physics experimentalists to teach proper laboratory techniques while fostering life-long mentor-

mentee relationships. SPS members learned to balance chemical equations, measure and mix glass samples, and roller quench and plate quench glasses for pelleting. The pelleting process is determined by the lithium content. Samples containing higher than 0.4 lithium oxide (0.33-0.35 in testing) will be roller quenched and pelleted. Dr. Bragatto is working with students to determine the ideal method of preparing the pellets for conductivity testing. Proposed ideas include: covering patches of the pellets with tape and placing them in a gold sputter, outlining paths with silver ink, and pouring melted glasses differently to avoid pelleting completely. Once a method is determined, SPS members will begin to perform electrical impedance spectroscopy measurements.

Dates	Objectives	Status	Officers Involved
January 14-25, 2019	Purchase required chemicals	Completed	Anne Ruckman
	and materials, Introduce		
	mentor/mentee SPS members		
January 14-31, 2019	Dr. Caio Bragatto's lecture on	Completed	Anne Ruckman
	ionic conductivity at Coe		
	College		
January 25-March 15,	Create glass samples, run MD	Completed	Martha Jesuit, Wataru Takeda,
2019	simulations for glass ratios		Anne Ruckman
February 15-May 30,	Dr. Steve Martin gives his	Completed	Anne Ruckman
2019	seminar on electrical		
	properties at Coe College		

- Project Modifications:
 - Installment of new equipment of Impedance and furnace for temperature was delayed. As a result, analysis tests were also delayed.
 - There was difficulty in finding a mutually convenient time for a trip to Iowa State University during the spring semester due to spring break and finals occurring at different times. The trip will occur during the summer because there are less conflicts for professors, students, and researchers.
 - SPS members learned a variety of sample preparation techniques as we modified our approach to preparing the glasses for testing. The time for students to learn and pursue alternative methods also delayed the conductivity tests.

Based on Wataru Takeda's theoretical model, we decided to produce $0.67 \text{ Li}_2\text{O}-0.33 \text{ B}_2\text{O}_3$ (Roller quenched) glass for further experimental testing.

- Personnel:
 - Dr. Steve Martin's Talk More than 25 SPS members and 5 non-members attended Dr. Martin's seminar on electrical properties of glasses. All participants networked and discussed graduate school research with him during breakfast and lunch.
 - Wataru Takeda Completed his mathematical model of the ionic conductivity of lithium borate systems, and submitted his publication
 - Martha Jesuit Completed the initial creation of the glass samples
 - Graham Beckler & Will Guthrie Completed training and are characterizing glasses
 - Seth White & Ethan Weber Completed training and are melting and running MD simulations on borate systems
 - Anne Ruckman Characterizing glass samples and directing SPS research and participation
 - Dr. Caio Bragatto Training, instructing, and leading SPS researchers through the glass forming process and analysis

- SPS connection:
 - Our project aims to start a connection with physics and engineering students at Iowa State University. We are strengthening our relationships and assisting students in networking with Dr. Steve Martin and Dr. Caio Bragatto. Locally, Coe College SPS members are learning safe, advanced practices for applied physics research while receiving support from their experienced mentors. Nationally, our chapter is contributing to the scientific works and studies of lithium clustering at high concentrations. Our novel research addresses lithium borate glasses, but may be witnessed in additional glass systems.

Updated Background for Proposed Project

Takeda, W., Wilkinson, C., Feller, S., & Mauro, J. (2019). Topological Constraint Model of High Lithium Content Borate Glasses. Submitted to *Journal of Non-Crystalline Solids*.

Wataru Takeda's model is being reviewed for publication, and our SPS members are relating the theoretical results to the experimental conductivity properties.

Description of Research - Methods, Design, and Procedures

Production: 5 SPS Members formed the following samples by melting them at 1000 °C for 1 hour:

- $0.1 \text{ Li}_2\text{O}-0.9 \text{ B}_2\text{O}_3$ (Plate quenched and Roller quenched)
- $0.2 \text{ Li}_2\text{O}-0.8 \text{ B}_2\text{O}_3$ (Plate quenched)
- 0.3 Li₂O-0.7 B₂O₃ (Roller quenched)
- $0.35 \text{ Li}_2\text{O}-0.65 \text{ B}_2\text{O}_3$ (Plate quenched)
- 0.5 Li₂O-0.5 B₂O₃ (Roller quenched)
- 0.67 Li₂O-0.33 B₂O₃ (Roller quenched)

Characterization: Each sample was crushed with a mortar and pestle into a fine powder between two aluminum foils. The glasses were then pressed into ¼" pellets by adding 1 drop of acetone, maintaining 2.5 kg of force for 30 seconds, and reducing the force to 1.85 kg for 5 minutes. The foils are used as electric contacts for impedance measurements.

Differential Scanning Calorimetry (DSC) is being run from room temperature up 600 °C at a pace of 10 K/min.

Initial Results

- Sample compositions completed as outlined in Description of Research.
- Sample characterization completed as outlined in Description of Research.

DSC analysis was completed for $0.3 \text{ Li}_2\text{O}-0.7 \text{ B}_2\text{O}_3$ (Roller quenched) and $0.67 \text{ Li}_2\text{O}-0.33\text{B}_2\text{O}_3$ (Roller quenched). DSC testing confirms our lithium borate samples' glass transition temperatures agree with accepted values in the literature⁷.

Statement of Next Steps

Plan for Carrying Out Remainder of Project (including Timeline)

Personnel:

• Graham Beckler & Will Guthrie – Continue characterizing glasses

- Seth White & Ethan Weber Continue melting the sample compositions outlined in Next Steps Production and running MD simulations on borate systems
- Anne Ruckman Continues characterizing glass samples and directing SPS research and participation
- Dr. Caio Bragatto Continues training, instructing, and leading SPS researchers through the glass forming process and analysis

Future Milestones:

- Dr. Caio Bragatto and SPS members will travel to ISU for SPS interaction and networking
- Researchers will finalize measurements and conduct analysis on density measurements, differential calorimetry scanning, impedance spectroscopy at low frequencies, and X-ray diffraction
- SPS members will draft the scientific publication of our findings
- Submit Final Report

Production: 3 SPS Members will form the following samples by melting them at 1000°C for 1 hour:

- 0.2 Li₂O-0.8 B₂O₃ --- Roller quenched
- $0.25 \text{ Li}_2\text{O}-0.75 \text{ B}_2\text{O}_3 --- \text{Plate quenched, Roller quenched}$
- 0.3 Li₂O-0.7 B₂O₃ --- Plate quenched
- $0.35 \text{ Li}_2\text{O}-0.65 \text{ B}_2\text{O}_3 --- \text{ Roller quenched}$
- 0.4 Li₂O-0.6 B₂O₃ --- Roller quenched

Dates	Objectives	Status	Officers Involved
March 1-April 30, 2019	Dr. Caio Bragatto gives his	In Progress	Anne Ruckman
-	seminar on glass conductivity		
	at Iowa State University		
March 15-May 30,	SPS members test glasses at	In Progress	Anne Ruckman, Martha Jesuit,
2019	Coe College and Iowa State		Wataru Takeda
	University		
May 31, 2019	Compile interim report	In Progress	Anne Ruckman
May 20-July 19	Summer research	In Progress	Anne Ruckman
	(glass preparation and		
	characterization)		
June 1-August 5, 2019	Create glass samples with	In Progress	Anne Ruckman
_	sample shaping process	_	
June 1-November 15,	SPS members analyze glass	In Progress	Anne Ruckman, Martha Jesuit,
2019	conductivity and submit	_	Wataru Takeda
	research paper		
December 31, 2019	Complete and submit final	In Progress	Anne Ruckman
	report		

Bibliography

[1] Varshneya, A. K. (1994). Fundamentals of Inorganic Glasses. Academic Press.

[2] Martin, S. W. (1991). Ionic Conduction in Phosphate Glasses. Journal of the American Ceramic Society, 74(8), 1767–1783.

[3] Montouillout, V., Fan, H., del Campo, L., Ory, S., Rakhmatullin, A., Fayon, F., & Malki, M. (2018). Ionic conductivity of lithium borate glasses and local structure probed by high resolution solid-sate NMR. Journal of Non-Crystalline Solids, 484(November 2017), 57–64.

[4] Charles, R. J. (1963). Some Structural and Electrical Properties of Lithium Silicate Glasses. *Journal of the American Ceramic Society*, *46*(5), 235–238.

[5] Voigt, U., Lammert, H., Eckert, H., & Heuer, A. (2005). Cation clustering in lithium silicate glasses: Quantitative description by solid-state NMR and molecular dynamics simulations. *Physical Review B* -*Condensed Matter and Materials Physics*, 72(6), 1–11.

[6] Takeda, W., Wilkinson, C., Feller, S., & Mauro, J. (2019). Topological Constraint Model of High Lithium Content Borate Glasses. Submitted to *Journal of Non-Crystalline Solids*.

[7] Feller, S., et. al (). Physical Properties of Alkali Borosilicate Glasses. In A. Wright, S. Feller, & C. Hannon (Eds.), *Borate Glasses, Crystals & Melts* (pp. 246-253). Oxford, UK: Alden Press.