

Advanced Glass Seal for Electrochemical Oxygen Separation-Compression Device

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Glass-based sealing materials are preferred for hermetic sealing of high temperature electrochemical devices due to flexibility in tuning the thermophysical properties by formulation chemistry. Ease of manufacturing and application are the other major advantages of glass seals and a number of glass systems have been reported as sealants for solid oxide electrochemical systems. Recent developments in sealing materials for oxygen separation and compression will be presented with focus on chemical and structural stability of electrode–seal and electrolyte–seal interfaces. The similarities and the contrasts between the sealing materials for oxygen compressor and the SOFC/SOEC will be discussed. Seal properties will be discussed with regards to constituent materials such as glass network formers, modifiers, additives, and intermediate oxides.

Nomenclature

T_g	=	glass transition temperature
T_m	=	melting temperature
Ω	=	Ohm (unit for electrical resistance)
A	=	ampere (unit for electrical current)
Pa	=	pascal (unit for pressure)
T_c	=	crystallization temperature
PO_2	=	partial pressure of oxygen
$SOFC$	=	solid oxide fuel cell
$SOEC$	=	solid oxide electrolysis cell
OTM	=	oxygen transport membrane
CSO	=	cerium samarium oxide
LCM	=	lanthanum calcium manganite
EVA	=	extravehicular activity
$ICP-MS$	=	inductively coupled plasma mass spectrometry
RT	=	room temperature
CRT	=	cooled to room temperature
DSC	=	differential scanning calorimetry
$HAADF$	=	high-angle annular dark-field

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I. Introduction

Hermetic sealing of electrochemical systems is required to promote efficient gas reduction and evolution reactions at active cell areas by separating and maintaining gas flow streams. Many planar electrochemical systems, such as solid oxide fuel/electrolysis cells (SOFC/SOEC) and oxygen/ionic transport membranes (OTM) utilize glass-based sealants over other sealant types due to the strong chemical bonding nature of these sealants.¹ The location of these sealants with respect to the cell stack and full system assembly is schematically pictured in Figure 1.¹ The system of further discussion is a type of planar OTM which is configured to compress pure or relatively pure oxygen. This system is designed for compression of oxygen for extravehicular activity (EVA) within space missions, using feed oxygen from existing electrochemical systems onboard the International Space Station.² This feed oxygen is to be compressed to 300 bar pressure in order to fill pressure tanks for proper storage.² The system is designed to pressurize in a single-step fashion, using a ceramic anode to reduce feed oxygen, a selectively-oxygen ion transporting ceramic electrolyte to allow diffusion of oxygen, and a ceramic cathode to oxidize the transported oxygen and subsequently allow gaseous oxygen to collect on one side of the cell stack. An electric potential drives the reduction/oxidation reactions while promoting oxygen diffusion only toward the cathode. Ceramic interconnects are used to provide structural stability to the cell stack and conduct electrons from the applied bias to the electrodes. Oxygen which collects at the cathode region of the stack is contained and subsequently pressurized. The details regarding this electrochemical system function, the materials which enable oxygen compression, as well as the fabrication processes for this particular planar cell stack design are discussed in ICES Submission 379 - Solid State Electrochemical Oxygen Separation and Compression.²

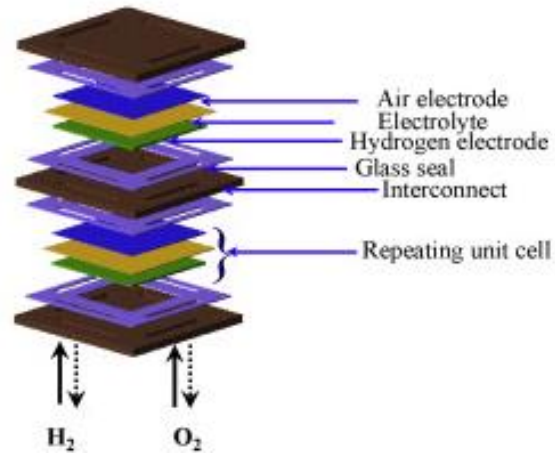


Figure 1. Schematic of electrochemical cell stack, highlighting locations of glass seals¹

There are various, stringent requirements for glass-based sealants within electrochemical systems, which Mahapatra and Lu review in great detail.¹ This paper will provide a brief overview of these requirements, which will lead into the discussion and reasoning behind a particular glass-based sealant tested for the aforementioned oxygen compression system. The sealant, sent to the University of Connecticut from collaborators, was analyzed in detail. This analysis was carried out to determine constituent elements and certain critical temperatures, such as the glass-transition temperature and melting temperature. Preliminary bonding tests were also carried out to analyze an important seal within the cell stack: the sealant/interconnect interface. To determine bonding capabilities, a bonding test was conducted at a specific temperature and mechanical load. A composite sealant/interconnect pellet was also developed to study interfacial reactivity between the glass sealant and interconnect material. This study on a candidate glass-based sealant to be used in an oxygen compression system highlights the feasibility of an efficient, planar solid oxide electrochemical oxygen compressor. The following is a description of glass-based sealants, which precludes the research findings on the analysis of the aforementioned sealant to be used for a solid state electrochemical oxygen compressor.

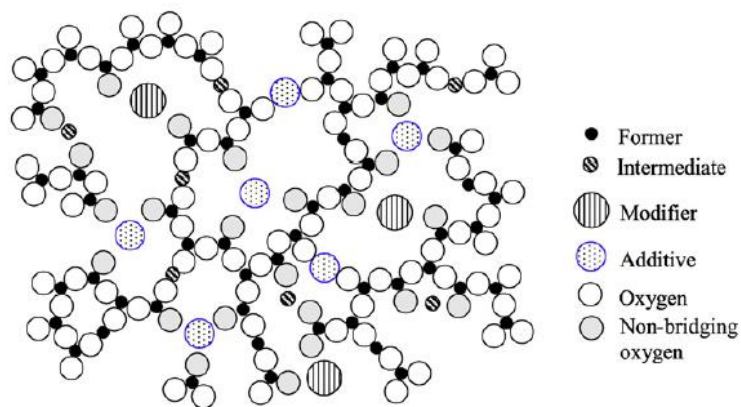
II. Glass-based Sealants in Electrochemical Systems

For proper sealing and overall efficient cell function, sealants must maintain stability over the entire duration of the system operation.¹ This includes stability and rigidity under various thermal cycles, mechanical stresses, and gaseous atmospheres.^{3,4} There are various types of sealants which have been suggested and used in electrochemical systems, however glass-based sealants have been found to provide optimum sealing and stability in system conditions.¹ These glass-based sealants must meet various requirements. In certain systems, sealants must also be stable under a range of oxygen partial pressures.⁵ These requirements and overall sealant properties are listed in Table 1.

Table 1. Properties and requirements for electrochemical system sealants^{1,6-8}

Properties	Requirements
Thermal	Thermal expansion coefficient at $9.5-12.0 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ Thermally stable for ~5000 h for mobile applications and for ~50,000 h for stationary applications at 650–900 °C cell operating temperatures
Chemical	Resistant to vaporization and compositional change in stringent oxidizing and wet reducing atmospheres at 650–900 °C (stability in high and low PO ₂) Limited or no reaction with other cell components
Mechanical	Withstand external static and dynamic forces during transportation and operation Resistant to thermal cycling failure during start-up and shut-down of cell stacks
Electrical	Electrical resistivity $\geq 10^4 \text{ } \Omega\text{cm}$ at operating temperature Electrical resistivity greater than 500 Ωcm between cells and stacks at nominal stack operating condition (0.7 V at 500–700 mA/cm ²)
Sealing ability	Sealing load <35 kPa Withstand differential pressure up to 14–35 kPa across a cell or stack Total fuel leakage <1% for the duration of the cell life
Fabrication flexibility	Flexible design, low processing cost, and high reliability

Glass-based sealants rely on various constituents which allow for tailorable sealant properties. Like all glasses, these sealants require network forming compounds, as well as network modifiers and intermediate oxides.¹ Network forming oxides, such as SiO₂ and B₂O₃, supply cations which form the basis of the glass network structure, acting as the center of polyhedra units. Modifiers are aptly named, as the addition of these oxides is used to modify certain thermal properties and create non-bridging oxygen atoms within the glass network. Similarly, intermediates can act as modifying oxides, specifically lowering the viscosity of the glass. They may also take part in the glass network, replacing network forming cations. Example intermediate oxides include Al₂O₃ and Ga₂O₃.¹ Oftentimes, other components called additives are included in glass sealant development to better tailor certain sealant properties such as coefficient of thermal expansion (CTE) or glass flowability.¹ These glass constituents, which atomically make up the glass network, can be visualized in Figure 2. With an understanding of glass constituents and the roles they play in the structure, certain glass sealant properties can be easily tailored to better design sealants for electrochemical systems.⁸

**Figure 2. Schematic of glass network¹**

As mentioned earlier, glass sealants must meet rigid requirements to aptly perform within an electrochemical system. The various constituents play vital roles in the sealant behavior and can be altered to provide better properties which match the system of question. The determination of these constituents with regards to the candidate glass sealant, as well as thermal property determination and bonding ability, will be discussed in the following experimental section. The interfacial stability between the sealant and ceramic interconnect was determined via post-test characterization of bonded test specimens. The interfacial stability is a key component of study for the candidate sealant as it would highlight any interfacial strength deficiencies or flaws in the developed seal.^{1,10} The stability must also be well-understood for future testing under high oxygen pressure, as the intended system must maintain sufficient sealing in an extreme pressure condition.

III. Candidate Sealant for Oxygen Compression

The candidate sealant was provided by American Oxygen LLC. (Salt Lake City, UT) in powder form. The sealant was to be used in preliminary bonding tests after the composition and thermal properties were determined. It is designed to seal similar ceramic components, which may avoid some challenges that surround sealing dissimilar

materials in other systems, such as sealing ceramics to metals.¹¹ Inductively-coupled plasma mass spectroscopy (ICP-MS) analysis and high-temperature x-ray diffraction (HT-XRD) were carried out to understand the composition and estimate the degree of sealant crystallinity. The sealant T_g and T_m were determined using

differential scanning calorimetry (DSC). The sealant was studied in an as-received state (powder) as well as after thermal treatment to understand morphology of the transitioned glass using scanning electron microscopy (SEM).

A. Inductively-Coupled Plasma Mass Spectroscopy Analysis

The constituents of the candidate glass sealant were determined using inductively-coupled plasma mass spectroscopy (ICP-MS). A 0.15 g sample of the sealant was digested, or dissolved, in trace metal grade HCl (1.5 mL) and HNO₃ (4.5 mL) solution and refluxed for 2 hours at 95 °C. The sample was then cooled and brought up to a final volume of 25 mL with de-ionized water for analysis. Two batches were used and the results are shown below in Figure 3. The first batch results showed mostly Ca, Mg, Na, and Ba with some Si. The relatively low amount of Si seemed incorrect since, of the five major constituents, Si is the only network former and would most likely make up the majority of the glass sealant network. For this reason, the second batch was created and tested. SiO₂ is relatively

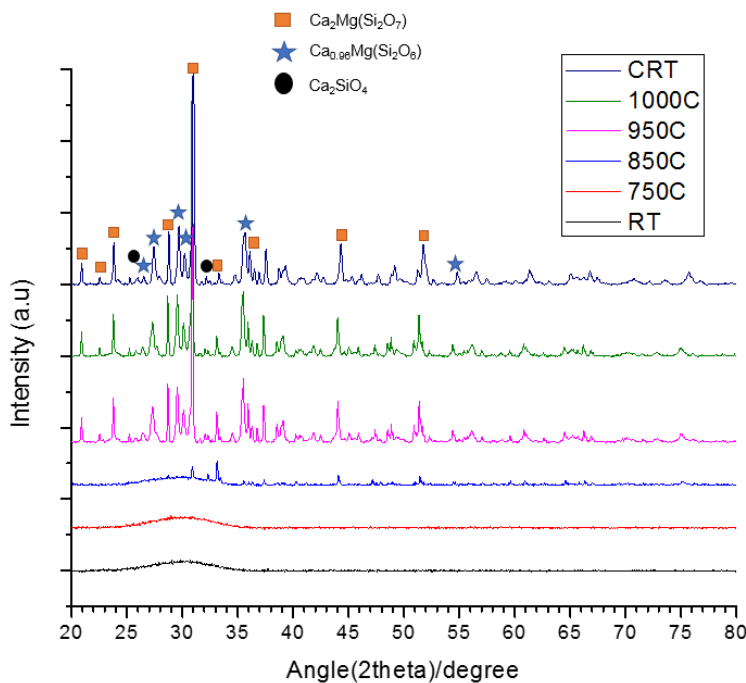


Figure 4. X-Ray Diffraction patterns taken at various temperature steps (temperature was held during scan). RT is room temperature and CRT is cool to room temperature.

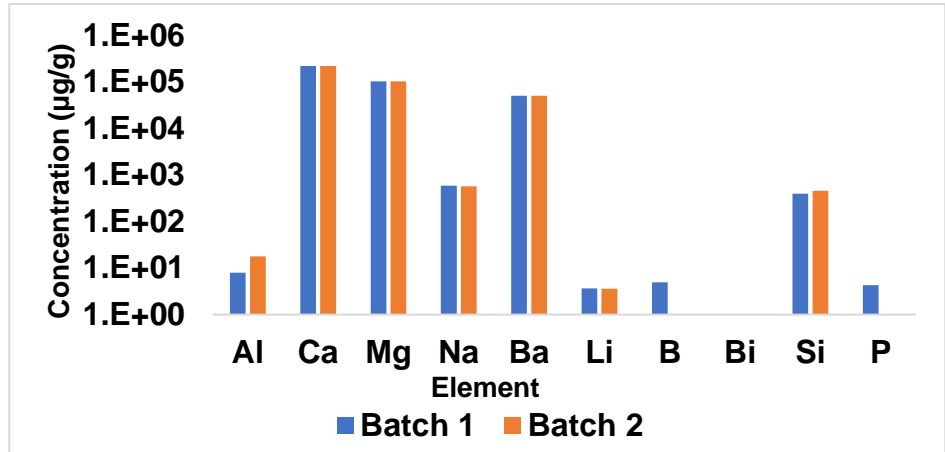


Figure 3. ICP-MS Results

difficult to digest so the second batch was refluxed for a longer duration of 4 hours to determine whether a higher concentration of Si would be observed. However, the relative concentrations of constituents did not change between batches (the second batch was not tested for Bi and P because these elements were deemed insignificant after the first batch result). It was then determined that SiO₂ would be too difficult to completely digest with the acid solution used and would require a stronger acid such as HF. The facilities used did not carry HF for digestion, so this result was left alone and further constituent analysis was carried out using x-ray diffraction.

B. X-Ray Diffraction

A small amount of the as-received glass sealant was analyzed using an x-ray diffractometer capable of high-temperature *in situ* analysis (Bruker). The sample was first scanned at room temperature (RT) and subsequently scanned after reaching temperature steps of 750, 850, 950, and 1000 °C before

being finally scanned once cooled down to room temperature (CRT). The resulting diffraction patterns for this *in situ* analysis are shown in Figure 4. At room temperature up to 750 °C, the glass sealant is amorphous as apparent by the broad diffraction hump at $2\theta=30$ degrees. At 850 °C, the onset of crystallinity can be observed with some early crystalline peak formation. This would elucidate the glass transition region prior to crystallization which the following section regarding differential scanning calorimetry will further explain. At 950 °C upwards, the sealant completely transforms to a crystalline structure composed of calcium magnesium silicates and calcium silicate, with other peaks which could not be accurately identified.

C. Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was used to better understand the transition from amorphous to crystalline structure observed in high-temperature x-ray diffraction. A small sample of as-received glass sealant was placed in a capillary tube for DSC analysis. The sample was heated to 1100 °C using a heating rate of 20 °C/min (note °C/min is equivalent to K/min) at ambient pressure. The T_g, determined as the

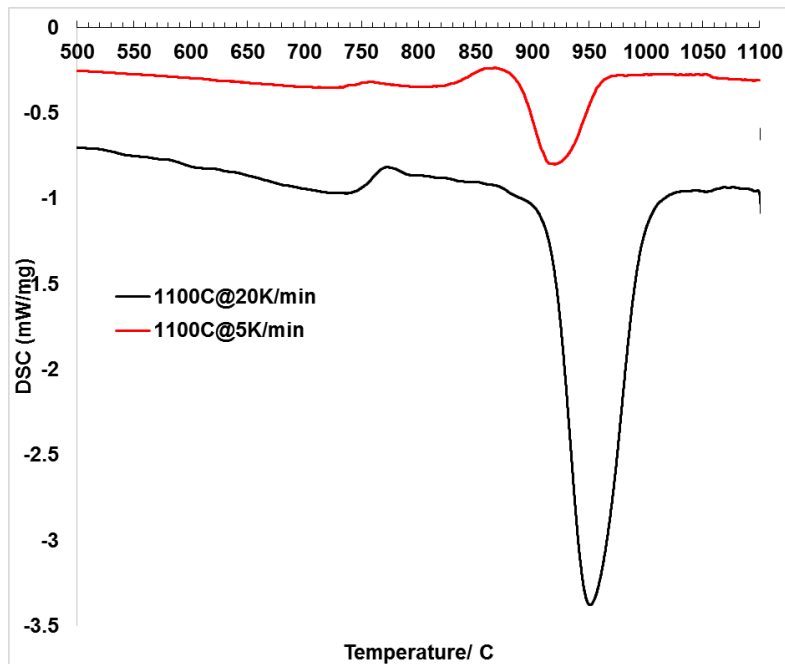


Figure 5. DSC scan of glass sealant heated to 1100 °C at 20 K/min (black) and 5 K/min (red)

endotherm at the onset of crystallization, occurred at 743 °C. The T_c, determined by the large exotherm, occurred at 950 °C. This result matched the onset of peak formation at 850 °C in HT-XRD as this temperature fell in the transition region.

To determine the effect of heating rate on T_g and T_c, another analysis was carried out at 5 °C/min up to 1100 °C. The endotherm corresponding to T_g and the large T_c exotherm both shifted when the sealant was heated at this slower rate. The T_g for this second analysis occurred at 726 °C and the T_c also shifted to a lower value of 920 °C. The results from both DSC experiments are presented in Figure 5. The shift in both temperatures denotes the effect of heating rate for glass sealants, which must be considered during bonding and ultimate integration in the system.

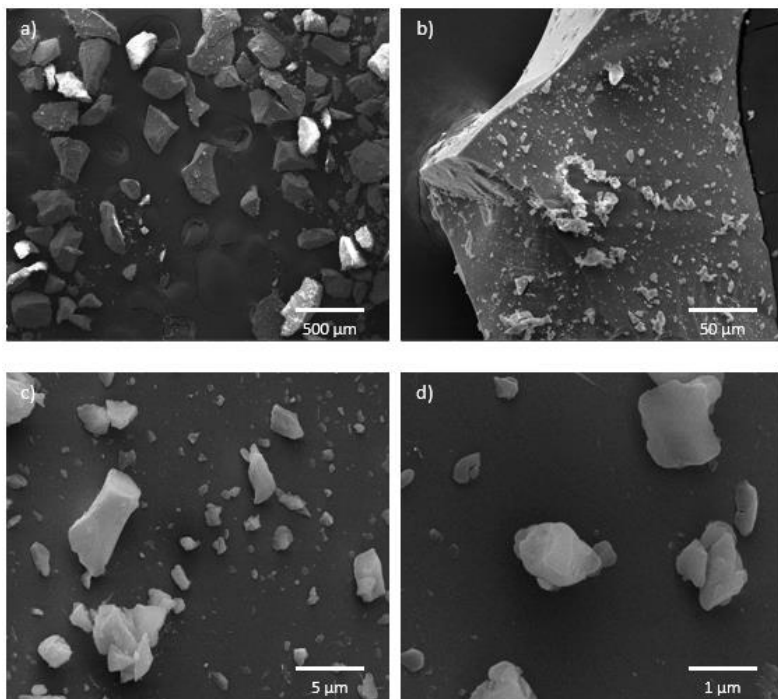


Figure 6. SEM of glass sealant particles on carbon tape substrate at: a) 100x magnification, b) 1,000x magnification, c) 10,000x magnification, and d) 50,000x magnification

D. Morphological Study

Scanning electron microscopy (FEI) was used to determine the morphology of the as-received glass sealant powder. A small sample of powder was placed on a

carbon tape substrate and any loose powder was carefully shaken off. The sample was coated in a thin layer of gold using a Denton sputter coater to ensure it was conductive for SEM analysis. The images taken using SEM are pictured in Figure 6.

The glass powder consisted of relatively large, 500 μm particles. These particles had many small particulates on them. These particulates ranged in size from $\sim 10 \mu\text{m}$ down to $1 \mu\text{m}$. The large particles were uniform both in size and in consistency of small particulates on the surface.

The preliminary morphological study was used to determine the room temperature structure of the glass sealant. In conjunction with HT-XRD, the glass sealant powder particles could be regarded as amorphous at room temperature.

IV. Glass Sealant/LCM/CSO Bonding Study

To determine sealant bonding capabilities, the sealant was pressed into uniaxial pellets and placed in between a pellet of the interconnect material and a pellet of the intended electrolyte material. The oxygen compressor will be designed using Lanthanum Calcium Manganite (LCM) ceramic interconnects and Samaria-doped Ceria (CSO) electrolytes within the cell stack.^{2,12-13} The LCM powder was provided by American Oxygen LLC in a calcined, pre-sintered state. The CSO electrolyte material was prepared via solid state synthesis. Ceramic powders of CeO_2 and Sm_2O_3 were added to a milling jar in stoichiometric amounts to achieve the formula $\text{Ce}_{0.85}\text{Sm}_{0.15}\text{O}_2$. The jar was filled with isopropanol and zirconia milling media and the solution was milled for 24 hours before being dried in a furnace at $90 \text{ }^\circ\text{C}$ until all isopropanol was evaporated. The resulting ceramic powder was pulverized using a mortar and pestle and pressed into pellets of $\frac{1}{2}$ " diameter. The glass sealant powder was similarly pulverized from its as-received state and formed into pellets of the same diameter. Prior to creating the pellet sandwiches, the LCM was sintered at $1350 \text{ }^\circ\text{C}$ for 4 hours and the CSO was sintered at $1550 \text{ }^\circ\text{C}$ for 4 hours. The sealant did not undergo any heat treatment prior to bond testing. These sintering conditions were provided by American Oxygen LLC. Sealing of the interconnects to the electrolyte and other cell components are imperative to system efficiency and separation of gaseous species.^{1,7} The following study revealed glass morphology after reaching elevated temperatures, bonding interfacial chemistry, and interfacial stability.

A. Bonding Test Set-up and SEM of Bonded Surfaces

The green-body glass sealant pellet was sandwiched between sintered LCM and CSO pellets atop an alumina plate. A silica glass slide was placed on top of the LCM and an alumina crucible weighted to 100 g was placed on top of the slide to provide roughly 8.5 kPa of pressure, which promoted bonding during thermal treatment. The LCM/Glass/CSO test set-up is shown schematically in Figure 7.

The test set-up was placed in a furnace at $950 \text{ }^\circ\text{C}$ for 1 hour using a heating/cooling rate of $5^\circ/\text{min}$. Based on DSC results, $950 \text{ }^\circ\text{C}$ is a sufficient temperature for the sealant to reach a glassy state and then crystallize after flowing to achieve good contact between both sintered pellet surfaces. After removal from the furnace, the two ceramic pellets appeared successfully bonded together with the glass sealant. The LCM/sealant/CSO bonded sample was then manually fractured and a piece revealing the cross-section of the bonded pellets was studied using SEM. The sealant/CSO and sealant/LCM interfaces are pictured in Figure 8. The low magnification image of the sealant/LCM interface (Figure 8b.) has a broad line of darker contrast due to the interface region being angled, which was caused by the manual fracture process. It should be noted that the CSO electrolyte (Figure 8c.) is not well-densified after sintering. However, the sintering process was not optimized since the objective of this test was to observe bonding between the CSO and sealant. Therefore, the porous CSO pellets were deemed acceptable to use for this preliminary test.

The two interfaces appear stable with good bonding across the majority of the sealant-contacted surfaces. Clear interfaces are apparent with no visual regions of secondary phase and little cracking/porosity at the interface. To better determine the chemistry of the bond and whether any interdiffusion of chemical species occurred or secondary phases formed during the bonding process, a cross-section was cut out of the LCM/sealant interface using Focused-Ion Beam

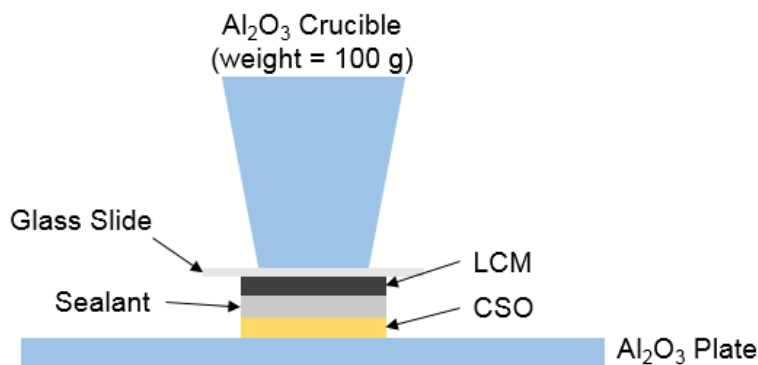


Figure 7. Schematic of bonding test set-up

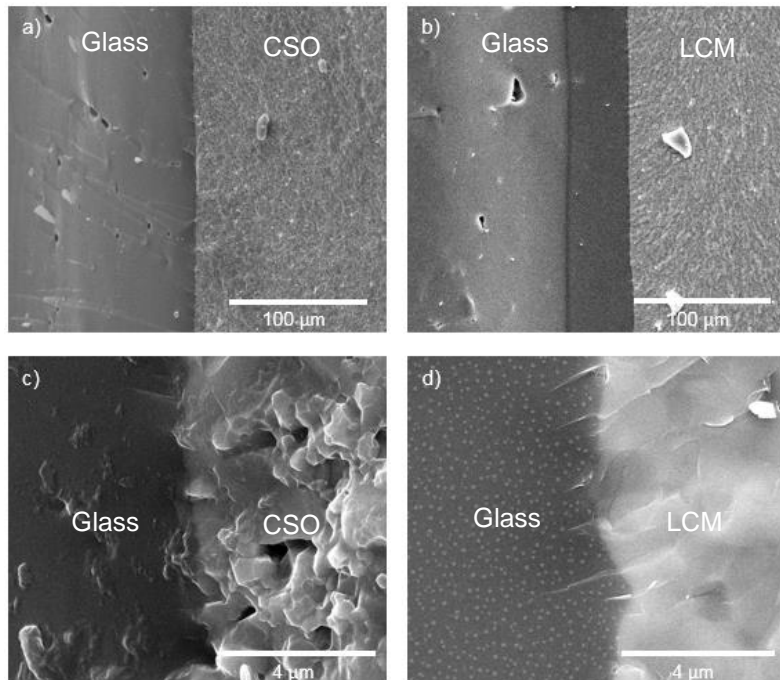


Figure 8. SEM of glass sealant/CSO interface (a and c) and glass sealant/LCM interface (b and d)

It should also be noted that the spots of darker contrast in LCM (Figure 9, left region of interface) are a Mn-based secondary phase within the LCM due to the Mn-rich stoichiometry of the material ($\text{La}_{0.4}\text{Ca}_{0.6}\text{Mn}_{1.0}\text{O}_3$). This phase has not affected key material properties of the interconnect although further analysis of this phase during cell operation may be needed to ensure the material is sufficient. This, however, is beyond the scope of this paper.

Elemental mapping was done at a location on the interface with good contact between LCM and sealant in order to investigate elemental distribution and any interdiffusion which may have occurred during bonding. This would provide better understanding of the phases which form under bonding conditions, which would provide a basis for further investigation into mechanical properties of these phases at high pressure. The region is chosen and subsequent mapping is shown below in Figure 10 with respect to a high-angle annular dark-field (HAADF) image taken to show interfacial contrast. Mapping was done for the following elements: Si, Ca, Mg, O, La, Mn, and Ba. Silicon is well-distributed within the glass sealant and no Si interdiffusion is observed. Calcium, however, is distributed in both structures but areas of high concentration are observed along the sealant/LCM interface. More work is needed to determine whether these Ca-rich deposits along the interface are either detrimental to the bond or whether they are key in the chemical bonding nature of the sealant. Magnesium, another sealant constituent, appears to interdiffuse preferentially into a region of LCM. This region is also Mn-rich, which may indicate the formation of a secondary Mg/Mn phase within LCM. The Mn-rich phase has already been observed in this particular LCM stoichiometry, however the presence of Mg may indicate a different phase. Similarly to the Ca-rich phase in the glass, the Mn/Mg phase appears adjacent to the sealant/LCM interface. This phase will also require more investigation to ensure it is not detrimental to sealant bonding capabilities.

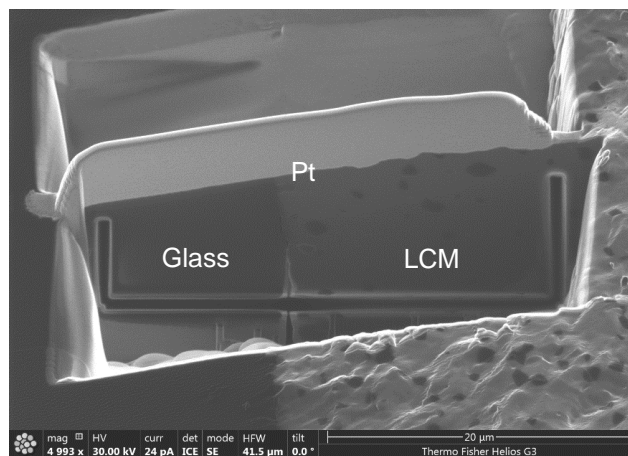


Figure 9. SEM image of FIB-milled cross-section for TEM analysis showing glass/LCM interface

(FIB, ThermoFisher) milling. The cross-section was then analyzed using the elemental mapping capabilities of a Transmission Electron Microscope (TEM, Talos™, Thermo Fisher Scientific™). The resultant elemental analysis is included in the following section.

B. TEM of Bond

A region of strong interfacial bonding between LCM and sealant was chosen for FIB milling and TEM analysis. The FIB-milled cross-section is pictured in Figure 9. The angle of the LCM/sealant interface surface, which caused a contrast difference in earlier SEM analysis, can be better observed in Figure 9. The milled section was first coated with Pt to protect the section surface and allow for sample lift-out after milling. During the milling process, a possible flaw in the interface may have propagated as the sample was thinned. However, the majority of the interface remained intact for subsequent TEM elemental mapping.

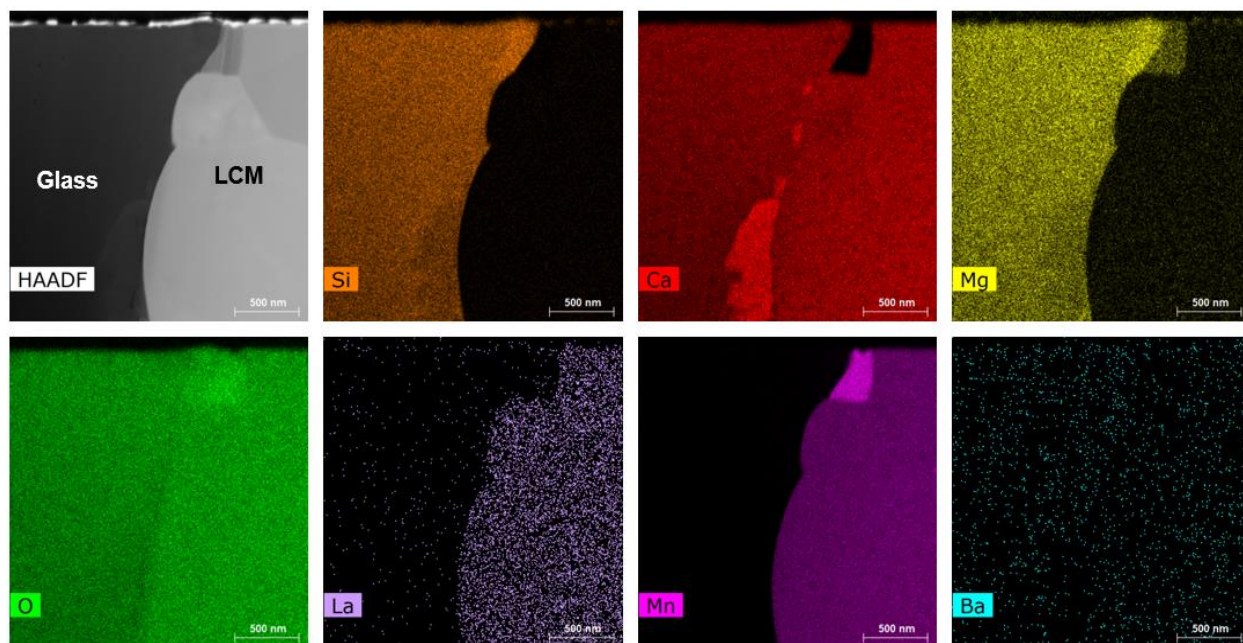


Figure 10. Elemental mapping of interface between glass sealant and LCM

Lanthanum is well-distributed within LCM, as to be expected. No clear interdiffusion of La into the glass sealant is observed.

V. Conclusion

A candidate glass-based sealant for an oxygen compressor system has been analyzed and preliminary bonding studies have been conducted. The ICP-MS results revealed the sealant is predominantly composed of Si, Na, Mg, Ca, and Al. Thermal characteristics of the sealant, mainly the T_g and T_c, were determined using DSC. The realm of glass-transition and crystallization were then used to design a bonding experiment in which a green-body pellet of the glass sealant was sandwiched between a sintered pellet of LCM and a sintered pellet of CSO. LCM is the planned interconnect material and CSO is the planned electrolyte material for an oxygen compressor system, which is discussed in ICES Submission 379 - Solid State Electrochemical Oxygen Separation and Compression.²

The preliminary bonding test revealed the given temperature, time, and pressure conditions were sufficient in creating a bond between the two ceramic pellets using the candidate sealant. TEM elemental mapping analysis of a cross-section taken at the sealant/LCM interface revealed the distribution of sealant and LCM constituents. This analysis revealed a Ca-rich region within the sealant, which preferentially formed along the interface. Whether this was a result of interdiffusion or reaction between LCM and the sealant requires further study. Another secondary phase, rich in both Mg and Mn, was found in LCM. This phase also preferentially formed along the interface. The presence of Mn-rich phases within LCM was expected, due to the Mn-rich stoichiometry of the LCM powder used, however the presence of Mg may also require further investigation. These early analyses and tests carried out on the candidate glass sealant are encouraging and prompt future work, which will analyze the sealant integrated within the oxygen compressor system.

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