Microstructural Studies of Strontium Based Alumino-Borosilicate Glasses for SOFC Applications

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Abstract—In the present study, the novel glass series (10+x) CaO-(10-x)MgO-10SrO-10B\textsubscript{2}O\textsubscript{3}-20Al\textsubscript{2}O\textsubscript{3}-40SiO\textsubscript{2} has been synthesized by melt quenching technique. For the stability and good efficiency of the solid oxide fuel cells (SOFCs) at high temperatures, hermetic sealants are required. Glass and glass ceramics offers promising sealant materials due to their adequate properties. Microstructural studies of the glasses (heat-treated at 850°C for 50 h) are done using Scanning Electron Microscopy (SEM) to gain insight of the glass.

Keywords- Solid Oxide Fuel Cell; Glass Sealant; Morphology; Scanning Electron Microscopy.

I. INTRODUCTION

Various research groups over the past few decades are making efforts to develop alternative sources of energy to save world from major threats like global warming, pollution and health hazards. Fuel cell technology is advancing rapidly as a promising alternate energy resource. Fuel cell converts chemical energy of fuels directly into electricity by an electrochemical reaction using oxygen and hydrogen as fuels. Solid oxide fuel cells are very efficient and clean source of energy having least material corrosion, high efficiency, high reliability, broad product range capability, high flexibility, environment friendly and electrolyte management problem [1-3]. Generally, two designs of SOFC’s are popular: planar and tubular geometries. Although, tubular design is most developed but due to its long current path around the circumference of the cell to interconnect, planar design is preferred because of its short current path resulting in high power density. During the fabrication, an appropriate sealing material is required for planar solid oxide fuel cells (pSOFC) [4-6]. The sealing prevents the mixing of gases and leakage losses at high working temperature of SOFC (800-1000 °C). Therefore, quality of the sealants must be high, since even small leakages of the air into the air will result in direct combustion leading to local overheating and thus, affecting and degrading the performance of the cell. Hence, the stringent requirements of the sealing material are high stability over a wide range, good mechanical compatibility with adjacent components, no harmful reactions with adjacent components, good wetting capability, air tightness, matching coefficient of thermal expansion and electrical insulation. Generally, glass and glass ceramics are considered as the most appropriate sealants because of their extensive properties like (i) good thermal expansion match (ii) good wetting ability during sealing (iii) ease to fabricate (iv) can avoid viscous flow and uncontrolled crystallization growth during the operation [7-9]. The present study elucidates the microstructural studies of the strontium based aluminoborosilicate glasses in order to gain insight and in-depth mechanism of the glasses. The scanning electron microscopy is a very powerful tool to investigate the chemical compatibility at the interface and the crystal growth or structure evolution processes. Also, for the stable glass sealants controlled crystallization is required as it leads to favorable microstructure. Thus, SEM analysis has been done in order to study about the crystal growth morphology [10-12].

II. EXPERIMENTAL TECHNIQUES

2.1. Preparation of Glass

The glass series (10+x) CaO-(10-x)MgO-10SrO-10B\textsubscript{2}O\textsubscript{3}-20Al\textsubscript{2}O\textsubscript{3}-40SiO\textsubscript{2} (x = 0, 2.5) chosen for the present study were prepared by taking required stoichiometric amounts of different constituent oxides or carbonates of 99.9% purity. Each batch was prepared by mixing an appropriate mole fraction of desired oxide ingredients in acetone medium using mortar and pestle. The powder obtained was melted in preheated furnace at 1550 °C in high resistance furnace. The melt was quenched in air using copper plates. The quenched glass was annealed at 500 °C in preheated furnace to remove the internal stresses from the glasses. These glass compositions with sample labels are shown in Table 1.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>CaO</th>
<th>MgO</th>
<th>SrO</th>
<th>B\textsubscript{2}O\textsubscript{3}</th>
<th>Al\textsubscript{2}O\textsubscript{3}</th>
<th>SiO\textsubscript{2}</th>
</tr>
</thead>
<tbody>
<tr>
<td>10CaMg</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>40</td>
</tr>
<tr>
<td>12.5CaMg</td>
<td>12.5</td>
<td>7.5</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>40</td>
</tr>
</tbody>
</table>

Table 1 Composition (Mo% ) Of Glass Constituents
2.2 X-ray studies
The X-ray studies of crystalline phases formed in the 200 h heat-treated glass were analyzed using high-resolution XRD in a Bruker D8 X-ray diffractometer. The XRD voltage was 45 kV and the beam current was 40 mA. The scan time per step was 600 s with CuKα radiation (\(\lambda = 1.5406 \, \text{A}^\circ\)). The phase identification was done by comparing the experimental diffractograms to standard patterns compiled by the International Centre for Diffraction Data (ICDD).

2.3 Microstructural studies
The interaction study was carried out by heating glasses at 850 °C for 500 hours before polishing mechanically and etching with dilute HF. Then, these glasses were analyzed under SEM (ZEISS EVO MA-10) to study the microstructures of the glasses.

III. RESULTS AND DISCUSSION

3.1 X-ray Diffraction
The X-ray analysis of both the glass samples 10 CaMg and 12.5 CaMg exhibits very sharp peaks of calcium orthosilicate phase \(\text{Ca}_2\text{SiO}_4\) (ICDD-00-006-0511) and enstatite phase \(\text{MgSiO}_3\) (ICDD-01-074-2017) crystalline phases. The formation of these phases can be possibly due to following mechanism:

\[
\begin{align*}
2\text{CaO} + \text{SiO}_2 & \rightarrow \text{Ca}_2\text{SiO}_4 \\
\text{MgO} + \text{SiO}_2 & \rightarrow \text{MgSiO}_3
\end{align*}
\]

3.2 Microstructural studies
The microstructural studies are very important to gain insight of the glass surface for efficient performance of the fuel cell. The scanning electron microscopy (SEM) is used to study the crystal growth morphology size, surface texture, roughness, and chemical composition of materials. During SOFC operation various chemical reactions can take place due to diffusion of ions at the interface, thus, affecting glass seal and interconnect interface which ultimately impedes fuel cell performance. Therefore, with this analysis the glass surfaces can be analyzed for sealing applications.

The microstructural studies has been investigated in (10+x) \(\text{CaO-} (10-x) \text{MgO-}10\text{SrO-}10\text{B}_2\text{O}_3\) glasses (x = 0, 2.5) in which SEM micrographs of glass samples 10 CaMg and 12.5 CaMg shows the formation of crystalline phases embedded in amorphous glass matrix which is in well agreement with X-ray analysis. Both the samples exhibits clean and smooth glass surface without any crack or pores in the crystalline phases formed. Hence, the favorable microstructure of the glasses is good for sealing applications.

IV. CONCLUSION
The microstructural studies has been investigated in (10+x) \(\text{CaO-} (10-x) \text{MgO-}10\text{SrO-}10\text{B}_2\text{O}_3-20\text{Al}_2\text{O}_3-40\text{SiO}_2\) glasses (x = 0, 2.5) in which SEM micrographs of glass samples 10 CaMg and 12.5 CaMg shows the formation of crystalline phases embedded in amorphous glass matrix which is in well agreement with X-ray analysis. Both the samples exhibits clean and smooth glass surface without any crack or pores in the crystalline phases formed. Hence, the favorable microstructure of the glasses is good for sealing applications.

REFERENCES


