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

Mandeep Kaur
Department of Applied Science
Baba Banda Singh Bahadur Engg College
Fatehgarh Sahib, Punjab, India

Vishal Kumar
Department of Physics
Sri Guru Granth Sahib World University
Fatehgarh Sahib, Punjab, India

Gurbinder Kaur
Department of Physics
Thapar University
Patiala, Punjab, India

**Abstract— In the present study, the novel glass series (10+x) CaO-(10-x)-MgO-10SrO-10$B_2O_3$-20$Al_2O_3$-40$SiO_2$ 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$^0$C for 50 h) are done using Scanning Electron Microcopy (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 corrrosion,high efficiency, high relaibility,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 $^oC$). Therefore, quality of the sealants must be high, since even small leakages of the fuel into 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 aluminoboro silicate 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-10$B_2O_3$-20$Al_2O_3$-40$SiO_2$ (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 at 1550 $^oC$ in high resistance furnace. The melt was quenched in air using copper plates. The quenched glass was annealed at 500 $^oC$ in preheated furnace to remove the internal stresses from the glasses. These glass compositions with sample labels are shown in Table 1.

Table 1 Composition (Mol%) Of Glass Constituents

| Sample Name | CaO | MgO | SrO | $B_2O_3$ | $Al_2O_3$ | $SiO_2$ |
|---|---|---|---|---|---|---|
| 10CaMg | 10 | 10 | 10 | 10 | 20 | 40 |
| 12.5CaMg | 12.5 | 7.5 | 10 | 10 | 20 | 40 |

*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 CuK$_a$ radiation (λ = 1.5406 A°). The phase identification was done by comparing the experimental diffractgrams to standard patterns complied by the International Centre for Diffraction Data (ICDD).

*2.3 Microstructural studies*

The interaction study was carried out by heating glasses at 850 ºC 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 $Ca_2SiO_4$ (ICDD-00-006-0511) and enstatite phase $MgSiO_3$ (ICDD-01-074-2017) crystalline phases. The formation of these phases can be possibly due to following mechanism:

$$2CaO + SiO_2 \rightarrow Ca_2SiO_4 \quad (1)$$

$$MgO + SiO_2 \rightarrow MgSiO_3 \quad (2)$$

*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.

Fig. 1(a) shows the microstructure of 10 CaMg glass after 500 h heat treatment at $850^0$ C. It exhibits a dense microstructure with crystalline phases, clearly indicating phase formation embedded in amorphous glassy matrix. This phase formation can be possibly due to formation of crystalline peaks of calcium orthosilicate phase $Ca_2SiO_4$ and enstatite phase $MgSiO_3$ phase which is confirmed by X-ray analysis also [13-15]. Hence, the composite exhibited controlled crystallization. Moreover, the glass is devoid of cracks, pores and pits which makes it suitable to be used as a sealant. Also, Fig. 1(b) shows SEM micrographs for second glass composition. The formation of spherical and needle shaped crystalline phases upon heating could be clearly seen on the glass surface. Also, both the compositions are depicting very clean and smooth surfaces devoid of unwanted delamination, cracks and pores. These results indicates that the glass samples will exhibit good bonding behavior with the interconnect and electrolyte also which is necessary in order to have minimum divtrification and CTE (coefficient of thermal expansion) mismatch between glass/electrolyte interface and glass/interconnect interface. Hence, the glass samples exhibited controlled crystallization, which is a favorable microstructure and further confirms the hermeticity to be used for sealing applications.

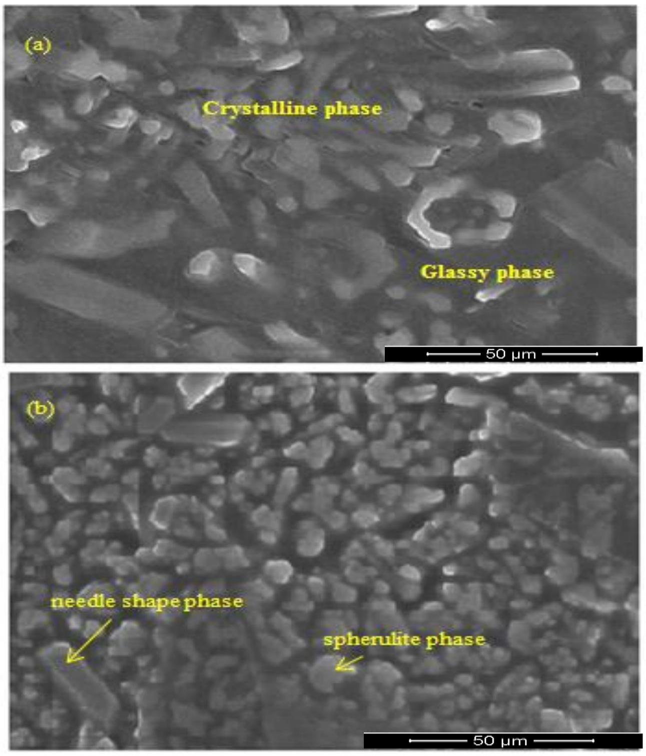


Fig. 1 (a) Microstructure of 10 CaMg glass heat treated at $850^0$ C (500 h), (b) Microstructure of 12.5 CaMg glass heat treated at $850^0$ (500 h)

## IV. CONCLUSION

The microstructural studies has been investigated in (10+x) CaO- (10-x) MgO-10SrO-10$B_2O_3$-20$Al_2O_3$-40$SiO_2$ 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.

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