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Low Temperature Glass Preparation

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ABSTRACT: A glass preparation technique for photonic applications with preparation method. This is a proven laboratory method for preparing silicates. Silicates are used as a base material for lasers, optical fibers etc. Classical silicate preparation requires very high temperature for preparation. The sol- gel technique offers low temperature preparation without melting and thus provides a better control over the microstructure. In the sol-gel route, glass hydrolyses and polymerizes at near room temperature ensuring better homogeneity in the resulting product. Aging changes the structure and phase of the gel due to hydrolysis and condensation and syneresis. During the sol-gel process, organic precursors are turned into a gel which is then transformed into a transparent and crack free solid glass upon densification due to sintering. The sintering is defined as a process to densify the sample upon application of high temperature of the order of 1100°C, which is very less in comparison to the normal route. The samples are then used for determining the parameters.

KEYWORDS: Gel, sintering, densification, sol, gel, aging.

I. INTRODUCTION

High purity glasses are much in demand in photonics, non-linear optical components, chemical sensors, semiconductor technology and many other related areas. During the sol-gel process, organic precursors are turned into a gel which is then transformed into a transparent and crack free solid glass upon densification due to sintering. The optical properties of Erbium doped in silicate matrix are being investigated for eye-safe laser wavelengths. by Obaton A.F. et.al.[1] for phosphate glasses. Ytterbium co-doped with Erbium exhibit lasing properties in blue and green regions of visible spectrum as cited by Jackel J.L. et. al. [2]. The conventional glasses require high temperatures for melting of powders. The sol- gel technique offers low temperature preparation without melting and thus provides a better control over the microstructure. In the sol-gel route, glass hydrolyses and polymerizes at near room temperature ensuring better homogeneity in the resulting product.[3- 8].

II. EXPERIMENTAL WORK

The sols were prepared by hydrolyzing tetraethyl orthosilicate (TEOS) with HCl acidified water. Calculated amount of dopant salts were poured in TEOS under vigorous stirring condition at room temperature. The molar ratio of TEOS: H₂O:HCl was 1: 14: 0.01. Hydrochloric acid was used as a catalyst. Very small quantities of HCl are enough to hydrolyse TEOS water mixture to a clear sol as it is a very strong acid. The pH of the sols was adjusted to 3.5 by addition of NH₄OH solution in it. The sols were cast into Petri dishes and covered with thin plastic sheets and left at room temperature. After several days, the sols changed to wet gels. The wet gels were aged at room temperatures for ten days and dried at 55° C for three weeks with a couple of pin holes on the cover and finally heat treated at temperatures > 900° C for sintering. This produced transparent, crack free glass. Different mole % (0.5%, 1.0%, 1.5%) Er₂O₃, 1.0 mole % Er₂O₃ with different mole % (0.5%, 1.0%, 1.5%) Yb₂O₃ and 0.5% mole % Al₂O₃ were prepared by using tetraethyl orthosilicate ErCl₃ 6H₂O, YbCl₃ 6H₂O, AlCl₃ 6H₂O, deionised water, HCl and NH₄OH.

III. METHODOLOGY

Polymeric Sol-Gel Technique

The main steps in the sol-gel process are (as in Figure 1):

1. Gelformation
2. Drying the gel

3. Consolidation of thegel

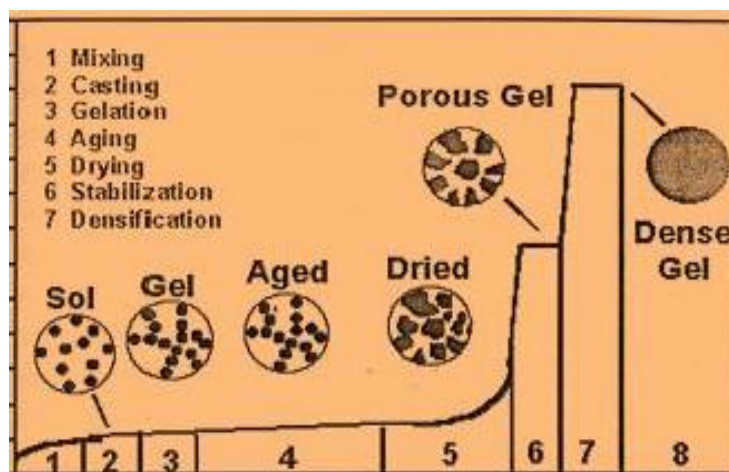
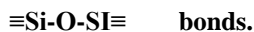


Figure 1. . Stages of sol-gel process

The dry gels are further heat treated to convert the porous xerogels into homogeneous transparent body free from porosity. During the sintering process, the driving process is the high surface energy of the gel . The pore elimination occurs through a viscous flow mechanism . The escape of organic volatiles from the gels may cause fractures during the initial heat treatments. Various factors influence the thermal behavior of the gels include water concentration, premature closing of the pores] causes gaseous residues. This causes expansion of the gaseous residues. At low temperatures, the consolidation is due to chemical reactions taking place inside the gel that are related to its structure. The chemical reactions may continue up to 800°C. Above 1000°C, physical sintering begins . The polymerization reaction may be classified into two steps.

Hydrolysis

Tetraethyl orthosilicate is hydrolysed by mixing with acidified water. The hydrolysis goes into completion depending upon the amount of water and catalyst present. The hydrated silica interacts or reacts with a TEOS molecule in a condensation reaction forming



Condensation

Linkage of additional Si -H tetrahedra occurs as a polycondensation reaction and eventually results in an inter-connected Si-O- Si network. The water and alcohol expelled from the reaction remains in the pores of the network. The water soluble metal salts (chloride, nitrate or acetate) are trapped in the pores of the network.

Polycondensation

The hydrolysis and polycondensation reactions initiated at numerous sites within TEOS and H2O solution as mixing occurs. When sufficient interconnected $\equiv\text{Si-O-Si}\equiv$ dimer bonds are formed as colloidal particles are condensed. The silica species link together, to become a three dimensional network. The physical characteristics of the gel network depend greatly upon the size of the particles and extent of crosslinking before gelation.

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There are many factors that influence the kinetics of hydrolysis and condensation. The factors affecting the kinetics of hydrolysis include temperature, nature and concentration of electrolyte, pH of the medium, nature of the solvent and alkoxide precursor. Hydrolysis and polycondensation may occur simultaneously.

Consolidation of Gel

The dry gels are further heat treated to convert the porous xerogels into homogeneous transparent body free from porosity. During the sintering process, the driving process is the high surface energy of the gel. The pore elimination occurs through a viscous flow mechanism. The escape of organic volatiles from the gels may cause fractures during the initial heat treatments. Various factors influence the thermal behavior of the gels include water concentration, premature closing of the pores causes gaseous residues. This causes expansion of the gaseous residues. At low temperatures, the consolidation is due to chemical reactions taking place inside the gel that are related to its structure. The chemical reactions may continue up to 800°C. Above 1000°C, physical sintering begins and the sample is obtained

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