PREPARATION OF $CaO-SiO_2-P_2O_5$ GLASS BY SOL-GEL ROUTE

A Thesis Submitted in Partial Fulfillment of the Requirement for the Degree of

Bachelor of Technology

BY

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13th MAY 2010 ABHISHEK MONDAL

CERTIFICATE

This is to certify that the thesis entitled, **"Preparation of CaO-SiO_2-P_2O_5 Glass by Sol-Gel Route"** submitted by Mr. Abhishek Mondal in partial fulfillment of the requirement for the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted in any other University/Institute for the award of any Degree or Diploma.

D. Sarkar

Ceramic Engineering

ABSTRACT

Typical $CaO-SiO_2-P_2O_5$ glass has been synthesized though sol-gel technique. Thermal analysis was carried out to evaluate the transformation temperature of glass. XRD analysis indicates the presence of several impurities along with amorphous phase.

CONTENTS

CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 Introduction of glass:

Glass is one of the oldest materials know to the mankind. It is usually a transparent or translucent material that has no crystalline structure yet behaves like a solid. The name 'Glass' is rather refers to the phase of the matter which holds equal importance as solid, liquid, gas or plasma. It is formed by freezing the liquid to a solid state without giving time to take structurally regular order. In other words, the atoms of a glass are held firmly in place like they are in a solid but are arranged randomly like those in a liquid. Hence glasses combine the properties of both liquids and solids but are also distinctly different from each. At the time when the glasses were considered as only inorganic materials, a well known definition of glass has been given "Glass is an inorganic product of fusion which has cooled to a rigid condition without crystallizing". The supercooled liquid constitutes a metastable phase [1]. It is nevertheless, in a state of internal equilibrium, being its free energy higher than that corresponding to the state of stable equilibrium. The free energy of the metastable liquid is kept in a local minimum, but not in an absolute minimum. This last case would imply the crystallization of the glass.

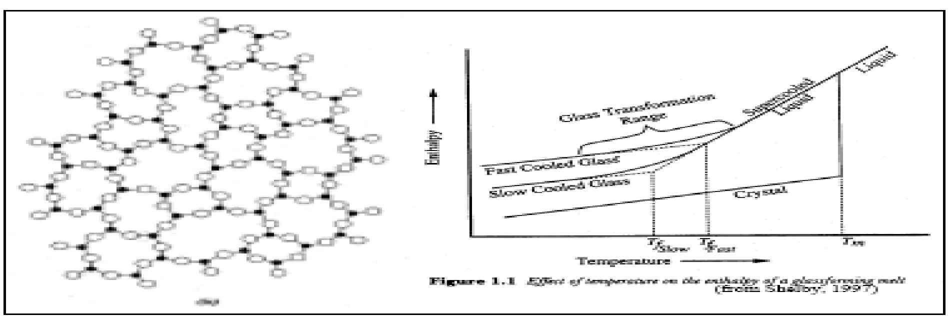

Figure 1.1 *Effect of temperature on the enthalpy of a glassforming melt* (from Shelby, 1997)

Figure 1. Structure of a glass and variation of enthalpy of glass forming melt with temperature.

When the liquid cools from the initial state (liquid), crystallization can occur at the liquidus temperature (T_m) in a discontinuous stepwise reduction of the specific volume followed by a milder contraction of the crystal. If the cooling happens to proceed sufficiently fast, the crystallization does not occur when T_m is reached, and one obtains a supercooled .

1.2. Background: Choice of Material

Among several glass compositions, the $CaO-SiO_2-P_2O_5$ glass has significant application in the field of bioimplant. This present glass has an ability to form strong chemical bonds with living bone as well as to achieve high mechanical strength. These glass-ceramics is useful for prosthetic applications in a number of load-bearing situations [2]. The $CaO-SiO_2-P_2O_5$ glass is the basic oxide system from which the ceramic bioactive materials, used us bone implants, are obtained. These materials are produced through melting of glasses und their controlled crystallization. This glass is used in clinical operations on bone defects and fractures may call for a filling material that also presents the ability to contribute to the healing process. Some of them are used as valuable bone substitutes such as artificial middle ear bones, alveolar ridge maintenance implants, artificial iliac crests and vertebrae. In this present work, the $CaO-SiO_2-P_2O_5$ glass has been prepared through sol-gel method. Finally the synthesized glass was characterized through XRD, DSC-TG analysis.

CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

2.1 Preparation of $CaO-SiO_2-P_2O_5$ glass

There are different routes by which $CaO-SiO_2-P_2O_5$ can be prepared which are given below:

1. Conventional melt-quenching method : In this method the batch is prepared , and grinded. The batch is heated upto a certain temperature at a fixed heating rate[2]. Then the batch is quenched at a fixed heating rate until it reaches the room temperature.

2. Freeze-extraction method : freeze-extraction is suitable for the production of the porous scaffolds in large scale. Time and energy can be greatly saved. In the freeze-extraction method, the solvent removed by extraction with non-solvent[3]. Therefore, the polymer would not dissolve because it is surrounded with non-solvent, hence it would be rigid enough to prevent pore collapse and formation of the surface skin.

3. One-pot method : one-pot synthesis route of simultaneous evaporation-induced self-assembly of Ca, P, Si and Fe sources and subsequent reduction in an H_2 atmosphere.

4. Sol-gel method : This process is a wet-chemical technique (also known as chemical solution deposition) which is widely used . Such methods are used primarily for the fabrication of materials starting from a chemical solution (sol, short for solution) which the precursor for an integrated network (or *gel*) of either discrete particles or network polymers. . In the sol–gel process, the metal alkoxide precursors could readily undergo catalysed hydrolysis and condensation reactions in the formation of a sol of metal oxide particles in nanoscale. The synthetic route via sol–gel is an efficient one, very easy and more convenient in the preparation of the $CaO-SiO_2-P_2O_5$ glass . Sol–gel derived glasses

have a much expanded compositional range of bioactivity over the glasses that are obtained from the traditional melting and casting process[4-8].

2.2 Advantages of CaO-SiO_2-P_2O_5 glass

The advantages of sol-gel derived glass are;[9]

(a) particulate as well as monolithic glass-like transparent materials can be formed without melting;

(b) distribution of cations are in a molecular scale, unique and uniform microstructures can be obtained in the liquid-liquid immiscibility zone;

(c) incorporation of certain cations in a particular co-ordination state can be achieved;

(d) suitable for making ultrapure glasses because many alkoxides are liquids which can be purified by distillation;

(e) highly flexible for depositing "glass" coatings at low temperatures;

(f) both the macroscopic homogeneity and submicroscopic homogeneity of glass prepared by the sol-gel process are considerably better; and

(g) homogeneous glass can be obtained at lower melting temperatures and in shorter melting times

CHAPTER 3

EXPERIMENT

3. Experimental Work

3.1. Preparation of Cao-SiO_2-P_2O_5 glass

0.1 M nitric acid was added to tetraethyl orthosilicate (TEOS) drop-wise to form a solution. The mixture was allowed to react for 30 minutes for complete acid hydrolysis of TEOS. $(NH_4)_2HPO_4$ and $Ca(NO_3)_2.4H_2O$ were added in sequence allowing 45 minutes for each reagent to react completely. Mixing was continued for 1 hr to allow complete hydrolysis. The resultant solution was cast into a cylindrical Teflon container and kept sealed for 10 days at room temperature to allow hydrolysis and polycondensation reaction to take place until a gelation. The gel was dried and the as-prepared powders were subjected to Differential Scanning Calorimetry- Thermal Gravimetric Analysis (DSC-TGA). The as-prepared powders were calcined at 1250 °C for 4 hours and were characterized by X-Ray Diffraction (XRD) using Cu-K_α radiations (λ=1.5409 nm).

3.2. Thermal Characterization of dried powder

The thermal behavior of the glass was studied by DSC-TG analysis. The thermo gravimetric (TG) curve was obtained by NETZSCH, which is heated starting from room temperature upto 1200^0 C with the heating rate of 10^0 C /min.

3.3. XRD analysis of prepared Glass

The phase analysis of the glass was studied using the room temperature powder X-ray diffraction with copper K-α (wavelength=1.54 A˚) radiation. Sample is scanned in a continuous mode from $20^0 – 80^0$ with scanning rate scale 0.04.

3.4 Flowchart

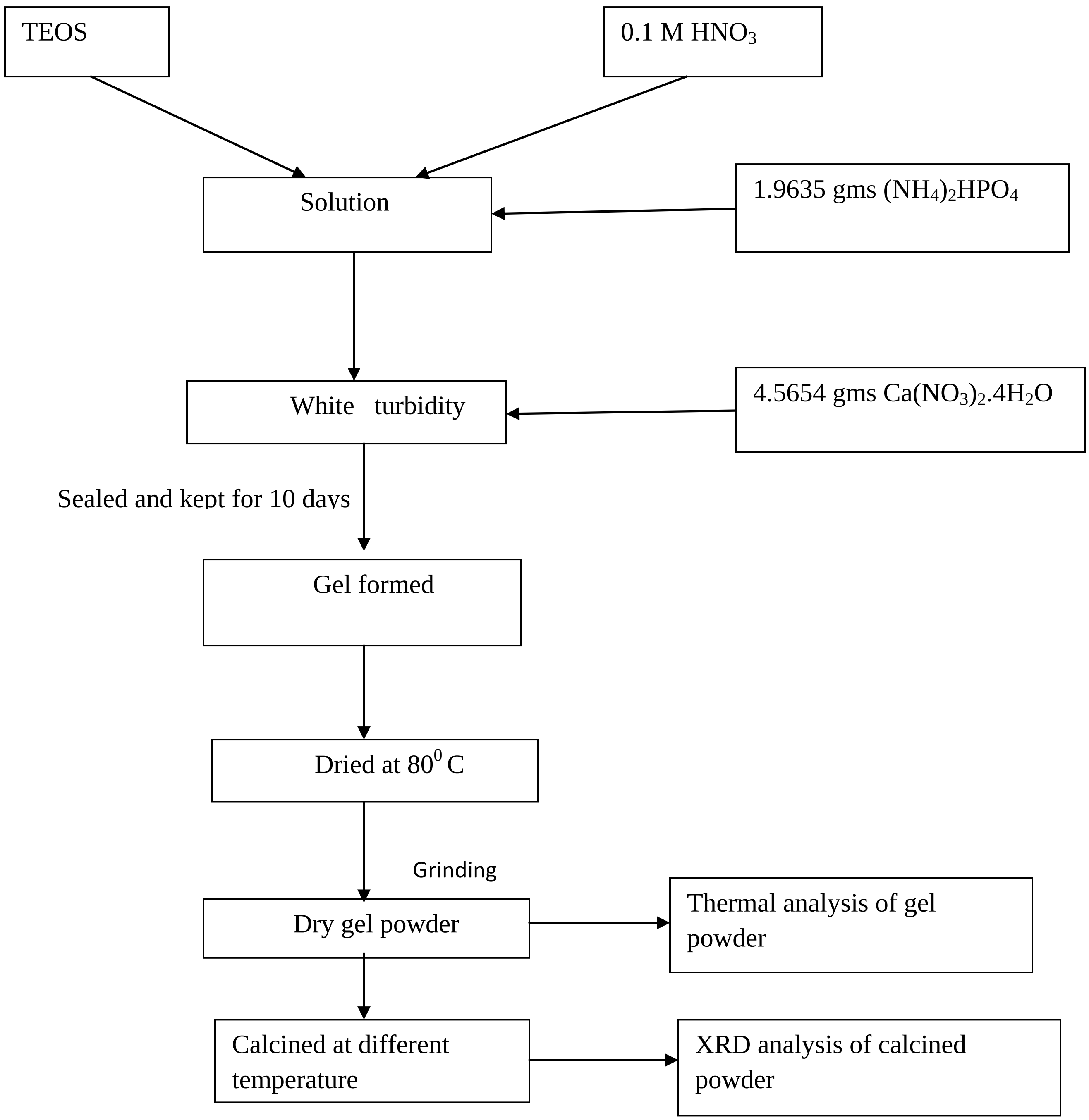

Figure 2. Flow chart for synthesis of CaO-SiO_2-P_2O_5 glass

CHAPTER 4

Result and Discussions

RESULT AND DISCUSSION:

4.1. Thermal behavior of sol-gel derived powder.

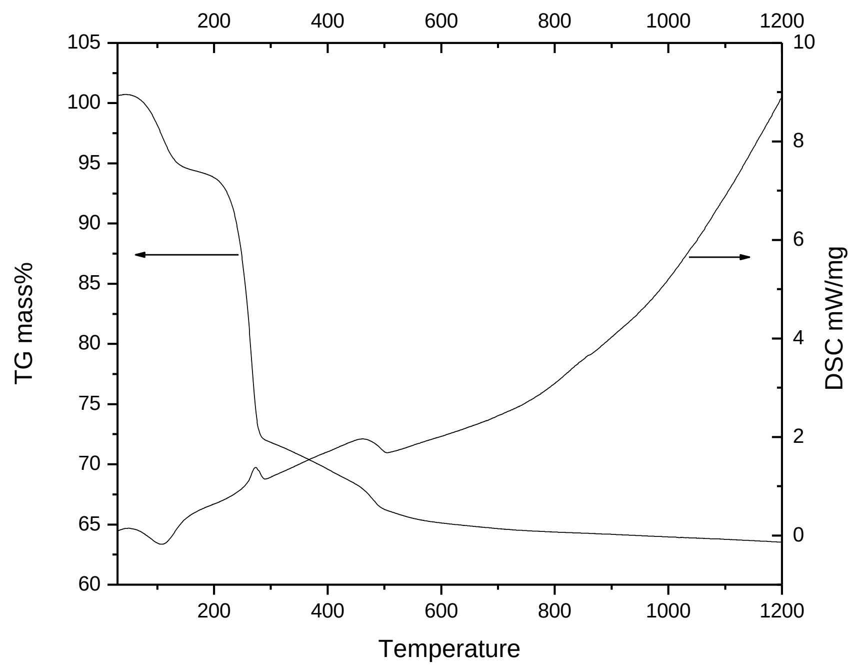

Figure 3. DSC-TG analysis of the dried gel powder.

Figure 3 exhibits the thermal behavior of dried powder. The weight loss (~6 wt%) could be observed at 100^0 C. Beyond this temperature, a significant weight loss (~22%) and an endothermic peak at 275^0 C was observed; associated with decompositions of precursors and residual water. The weight loss was linear beyond 800^0 C and no existence of exothermic and/or endothermic peak. Based on this thermal analysis, the temperature was selected for calcinations and finally the XRD analysis was carried out to justify the formation of the selective glass.

4.2 X-ray diffraction analysis:

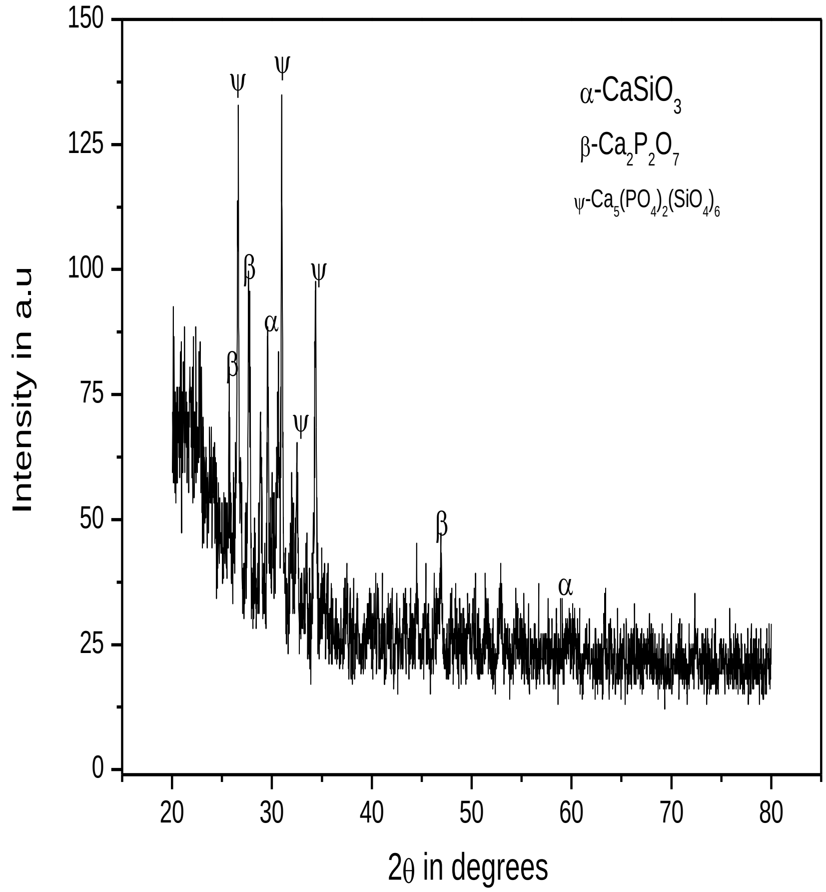

Figure 4. X-ray diffraction analysis of the calcined powder.

The above figure 4 shows the XRD analysis of the calcined glass. Different impurities phases could be observed as crystalline . The highest peak contains the composition $CaSiO_3$ phase, which is denoted by Ψ in the plot. The other peak which are represented by α and β are identified to be the phase $Ca_2P_2O_5$ and $Ca_5(PO_4)_2(SiO_4)_6$ phase respectively which are the impurity present in the glass composition.

CHAPTER 5

CONCLUSION

CONCLUSION

1. Thermal analysis suggests that 1000^0 C is the ideal temperature for the formation of CaO-SiO-P_2O_5 of glass.

2. The glass could be prepared through sol-gel route with having other crystallization impurities like $Ca_2P_2O_5$ and $Ca_5(PO_4)_2(SiO_4)_6$.

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