RESEARCH ARTICLE

FTIR Measurements of SiO₂ Glass Prepared by Sol-Gel Technique

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Abstract: The sol gel route allows powder-less processing of glasses and ceramics. Sol gel chemistry provides mild conditions so that organic species can be mixed with the inorganic precursors, leading to the formation of hybrid organic-inorganic systems. In the present work silica glasses were prepared by sol gel technique. The structural properties of silica glasses has been investigated with Fourier Transform Infrared Spectroscopy (FTIR), since Infrared spectroscopy may probe the nature of the bond between the metal cation and its surrounding oxygens and can distinguish the bonding between metal cations with oxygen.

Keywords: Glass, Sol-gel synthesis, Structural properties, Tetraethoxy silane, Fourier Transform infrared spectroscopy

Introduction

Sol gel is an attractive synthetic method to metal oxide materials applied in a wide variety of fields such as ceramics, sensors, catalysts, optics, electronics and thin films¹⁻³. The sol-gel method has several advantages such as high purity, ultra homogeneity, lower processing temperatures, and most significantly the possibility of making glasses of new compositions. The sol-gel technique has become quite popular during the past few years.

Sol-gel derived silica-titania gel films provide considerable interest⁴⁻⁶ since they enable fine control of refractive index and thickness for optical applications. For a planar waveguide application, the basic condition is that the guiding layer must have a refractive index higher than the substrate and cladding optical media. Single mode waveguide on fused silica (n=1.45) or soda lime substrates (n=1.51) requires a thickness of the guiding layer typically in the range of 0.2 to 5 mm, depending on the refractive index of the layer itself (in the range 1.65 to 1.512). Currently, the synthesis of SiO₂ by the sol-gel method has proven to be a very useful tool in the fields of environmental purification, solar energy cells, photocatalysts, gas sensors, photoelectrodes and electronic devices.

Experimental

The preparation of the sol gel glass specimens is similar to the method given by Vinoy *et al.*⁷ The sol gel glass specimens were prepared by mixing TEOS (Aldrich, 99.9%), ethanol (AR grade 99.9%), double distilled water and HCl (Nice chemicals) in the ratio 1: 4: 14: 0.01 respectively. TEOS and ethanol were magnetically stirred thoroughly till both were in well-mixed state. To this well-mixed solution the remaining water was added in which the desired acid was mixed. Again the solution was magnetically stirred to get a clear solution. The sols were cast in polypropylene dishes and were sealed to avoid intercalation of external impurity. The gels were aged for one month at room temperature to obtain the sol-gel. Lifetime may be shortened if the water content is not reduced to very low concentrations during drying and densification processes⁸. Hence, the samples taken in silica crucibles were sintered in a Muffle furnace at 600 °C for 3 h and then the furnace was cooled to room temperature at a rate of 0.5 °C per minute. The highest temperature required to obtain a silica glass prepared by sol gel method is about 1000 °C, which is lower by about 1000 °C compared to the temperature required for the melt-quenching process⁹.

Any structural changes in the samples prepared have been studied in the IR fundamental region (4000-400 cm³) on a Perkin-Elmer 1710 spectrometer using the Perkin-Elmer Infrared Data Manager using the KBr pellet method. Glass pellets for the infrared investigation were prepared by mixing thoroughly ~ 4 mg glass powder with ~ 200 mg dried KBr powder and compressing the resulting mixture in an evacuable die under 10 ton pressure for 5 min in order to yield transparent discs suitable for mounting in the spectrometer. The infrared spectrum were recorded in the range 400-4000 cm⁻¹.



Figure 1. Infrared spectrum of sol-gel glass

Results and Discussion

The absorption band at 3444 cm⁻¹ and 1639 cm⁻¹, showing that only a small amount of water is present in the glass, can be attributed to the stretching and bending vibration of either free OH groups or free H₂O molecules. The water has no substantial effect on the structure of the glass¹⁰. Metal alkoxides of Si(IV) are reported¹¹ to show absorption bands attributed to v(M-O) stretching modes around 800 cm⁻¹, respectively. Therefore IR peaks of TEOS at 794 cm⁻¹ in Figure 1 are assigned to v (Si-O)) stretching vibration. The very broad strong peak at 1083 cm⁻¹ can be ascribed to composite of C-O stretching of TEOS and ethanol¹².

It is also often reported¹³⁻¹⁵ that bands within the range 900-1000 cm⁻¹ are composite features of Si-OH species. The strong band at the frequency of ~950 cm⁻¹, therefore, is assigned to stretching vibration of Si-OH. The peak near 450 cm⁻¹ and a low frequency peak near 576 cm⁻¹ is assigned to Si-O-Si out of plane bending and Si-O-Si stretching modes respectively.

Conclusion

The SiO₂ glass was successfully synthesized by sol-gel method. FTIR spectroscopy showed the Si-O, Si-O-Si bonds were formed. The IR spectra showed that our synthesized material have similar and even better features than the reference material employed.

References

- 1. Kessler V G, Spijksma G I, Seisenbaeva G A, Hakansson S, Blank D H A and Bouwmeester H J M, *J Sol-Gel Sci Technol.*, 2006, **40(2-3)**, 163-179; DOI:10.1007/s10971-006-9209-6
- 2. Kayan A, Hoebbel D and Schmidt H, J Appl Polym Sci., 2005, 95(4), 790-796; DOI:10.1002/app.21315
- 3. Zhang X H, Yang J W, Zeng Z H, Chen Y L and Wang H H, *Acta Polym Sin.*, 2006, 750-755.
- 4. Whang C M and Lim S S, Bull Korean Chem Soc., 2000, 21, 1181.
- 5. Orignac X, Barbier D, Du X M and Almeida R M, Appl Phys Lett., 1996, 68, 895.
- 6. Matsuda A, Metsuno Y, Katayama S and Tsuno T, J Mater Sci Lett., 1997, 8, 902.
- Thomas Vinoy, Jose Gijo, Jose Gin, Biju P R, Rajagopal S and Unnikrishnan N V, J Sol-Gel Sci Technol., 2005, 33(3), 269-274; DOI:10.1007/s10971-005-6376-9
- 8. Thomas Vinoy, Ph. D Thesis, Mahatma Gandhi University, Kottayam and the references therein, 2002.
- 9. Yamane Masayuki and Asahara Yoshiyuki. 'Glasses for photonics'. Cambridge, University Press, Cambridge, UK., 2000, 271.
- 10. Scholze H, Glass Ind., 1966, 47(11), 622.
- 11. Bradley D C, Mehrotra R C and Gaur D P, Metal Alkoxides; Academic Press: New York, 1978, 116-122.
- 12. Matos M C, J Non-Cryst Solids, 1988, 232.
- 13. Whang C M and Lim S S, Bull Korean Chem Soc., 2000, 21, 1181.
- 14. Aizawa M, Nosaka Y and Fujii N, J Non-Cryst Solids, 1991, **128(1)**, 77-85; DOI:10.1016/0022-3093(91)90778-5
- 15. Schraml-Marth M, Walther K L, Wokaun A, Hamdy B E and Baiker A, *J Non-Cryst Solids*, 1992, **143**, 93-111; DOI:10.1016/S0022-3093(05)80557-5