

EDFA Materials

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Abstract - High purity glasses are much in demand in photonics, non-linear optical components, chemical sensors, semiconductor technology and many other related areas. 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. 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 amplification properties offered by Erbium doped Glasses at 1.6 μ m are highly sought after in photonics. This property is used in Erbium doped Fiber Amplifiers (EDFA). The loss encountered by the propagating wave in the optical fiber is compensated for by the EDFA. Samples of various stoichiometry doped with Erbium, Ytterbium and co-doped with Aluminum were prepared. XRD of samples sintered below 1100° show absence of crystalline structure. DTA/TGA show that the weight loss is due to removal of hydroxyl ions. FT-IR Spectra was observed using the KBr pellet method. Micro Raman spectra were measured using Argon laser as exciting source.

keywords - erbium, glasses , photonics, KBr pellet, sol-gel

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- 15].

2. EXPERIMENTAL

Sample preparing and analysis

Sample Preparation

Analytical Grade Tetraethyl Orthosilicate (TEOS), ErCl₃.6H₂O, YbCl₃.6H₂O, HCl, NH₄OH and de-ionized water were the starting materials for sol-gel glasses [4]. Molar ratios of 1 : 14 : 0.01 were taken for TEOS : H₂O : HCl and magnetically stirred at about 50 °C (Remi make Magnetic Stirrer cum temperature controlled hot plate) till a clear liquid is formed . At this time, calculated and carefully weighed dopants (Sartorius, Semi Mico Analytical single-pan electronic balance) were added and stirred again to mix the salts. Strips were used to check the pH of the solution and NH₄OH was added to yield a pH of 3.5 (using a Microlit micro pipette) . The sols were poured into Tarson Petri dishes and covered with polythene sheets tied tightly with rubber bands with several pin-holes on the cover sheet to allow moisture to escape from the sample. They were then kept for seven days at room temperature and then baked in a digitally controlled oven for three weeks at 55°C for aging. Aging changes the structure and phase of the gel due to hydrolysis and condensation and syneresis .The samples are then sintered in a programmable electric furnace (Eurotherm make) in pre decided temperature steps upto 1100 °C. This produced transparent glass.

Analytical Techniques

XRD was carried out in (10° to 70°) 2 θ range using a Philips (PW 1710 diffractometer with CuK α radiation ($\lambda = 1.5405\text{\AA}$)). TGA/DTA analyses were carried out in (Pyris Diamond TGA/DTA analyzer). The FT-IR were measured with the help of Spectrophotometer (Nicollet, NEXUS 870).

3. RESULTS AND DISCUSSIONS

Visual Characteristics

The samples were pink in color after sintering. The samples looked translucent before sintering and became transparent after sintering.

Structural characteristics

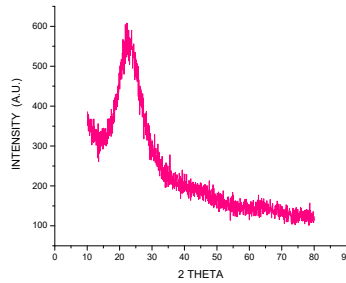
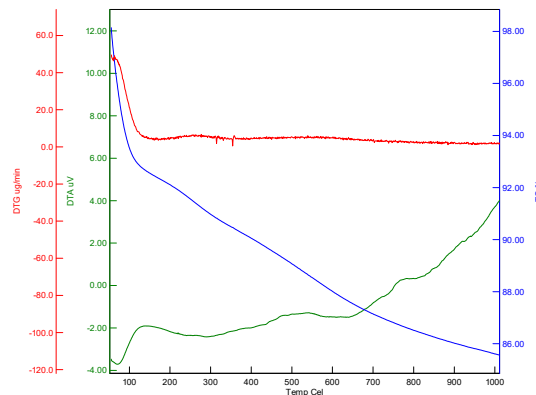


Figure 1: The XRD patterns of 1.5% Er₂O₃ 0.5% Yb₂O₃ silica gel

Fig. 1 shows XRD pattern of 1.5% Er₂O₃ 0.5% Yb₂O₃ silica gel XRD was carried out in (10° to 70°) 2θ range using a Philips (PW 1710 diffractometer with CuK_α radiation (λ = 1.5405Å). It is observed that there are no sharp peaks at sintering temperature of 950 °C and thus shows glassy nature of the silica gel .



Thermal Characteristics

Fig. 2 : The DTA/TGA/DTG patterns of 1.5% Er₂O₃ 0.5% Yb₂O₃ silica gel

In Fig. 2 the TGA curve shows weight loss as temperature increases. Initially the fall is very fast for temperatures below 100 °C which is attributed to removal of water. The organic precursors are decomposed which is manifested as weight loss around 550 °C . The TGA curve also shows a kink at this temperature. The final stage of weight loss is probably attributed to condensation which essentially liberates a small molecule of water in this case.

Fourier Transform Infrared Spectra

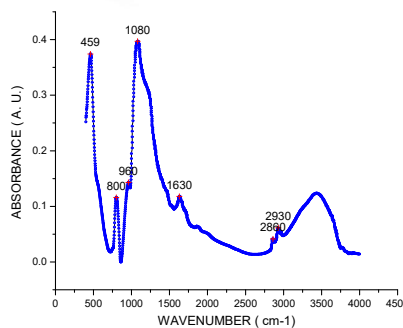


Fig.3 FT-IR of Er₂O₃ 0.5% Yb₂O₃ silica gel

Fig. 3 shows FT-IR pattern of 1.5% Er₂O₃ 0.5% Yb₂O₃ silica gel. IR spectra of the various silica gels were observed by the KBr pellet method in the range (4000-400 cm⁻¹) using a FT-IR Spectrophotometer (Nicollet, NEXUS 870). There are distinctive peaks in the spectra at 459 cm⁻¹, 800 cm⁻¹, 960 cm⁻¹, 1080 cm⁻¹, 1630 cm⁻¹, 2930 cm⁻¹ and a broad peak at 3438 cm⁻¹ for the silica gel .The peak at 459 cm⁻¹ is due to the bending vibration of Si-O-Si . The peak at 800 cm⁻¹ is assigned to the symmetric stretching vibration of Si-O-Si .The peak at 960 cm⁻¹ is due to stretching of Si- OH . A very intense peak at 1080 cm⁻¹ is due to the asymmetric stretching vibration of Si-O-Si . The broad peak around 3438 cm⁻¹ is due to silanol .

Micro Raman Spectra

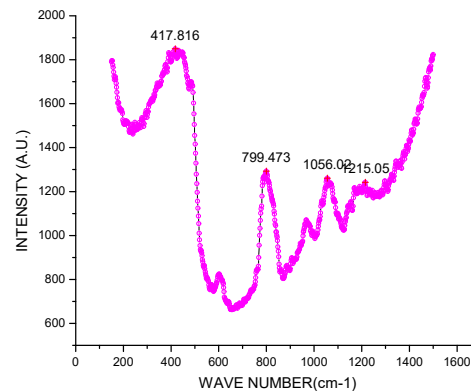


Fig. 4: The Micro Raman Spectra patterns of 1.5% Er₂O₃ 0.5% Yb₂O₃ silica gel

Fig. 4 shows Micro Raman Spectra of the silicate gels using a Renishaw Microspectrometer, RM1000. The 514.5 nm of an Ar⁺ laser had been used to excite the Raman scattering. The lasers output power was 30 mW. The Micro Raman pattern of 1.5% Er₂O₃ 0.5% Yb₂O₃ silica gel. The Micro Raman peaks were observed at 417 cm⁻¹, 799 cm⁻¹, 1056 cm⁻¹, 1215 cm⁻¹. The group and finally the peak at 1630 cm⁻¹ is due to adsorbed water. [16-20] 430 cm⁻¹ is allotted to motion of oxygen, popularly called the symmetric ring breathing mode. Symmetric Si-O-Si stretching mode is represented by 800 cm⁻¹. The band at 1056 cm⁻¹ due to the C-H stretching vibrations of un-hydrolyzed species. The 1215 cm⁻¹ band arises due to Si-O asymmetric stretching vibrations. [16-25]

4. ACKNOWLEDGMENTS

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