Absorption Peaks of Erbium Silicates

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Abstract **- Low processing temperature, homogeneity and high purity are the advantages one attains through hydrolysis and condensation of metal alkoxides to derive oxide glasses when compared with conventional melting techniques. The main steps in the sol-gel technique are the gel formation, drying of the gel and consolidation of the gel which are governed by the stoichiometry, pH, physical conditions, and subsequent heat treatments. An attempt was also made to explore the effects of varying the aging time of the gels to three years for chemical stability of the glasses produced. Earlier researchers have studied Titania nanocrystalline powders have reported similar studies on fiber– optic sensors with fluorescein isothiocyanate (FITC) pH sensitive dye. The FT-IR spectra of the stored erbium doped silicate xerogels are studied extensively. The various IR Spectra, in the range (4000- 400 cm^{-1}) of erbium doped stored silica gels as a function of sintering temperature are determined. All measurements were carried out by Nicolet, NEXUS 870 Spectrophotometer by using the KBr pellet method. The variation of Erbium doped IR Spectra as a function of sintering temperature reveal many interesting results. The maximum absorption peaks around 3400 and 1630 cm^{-1} are observed for unsintered gels and gradually decrease on increasing sintering temperature thereby suggesting escape of molecular water. The structural indication of Si-OH bonds is by band at around 960 cm^{-1}. Defect structure is assigned to bands around 750 and 550-600 cm^{-1} in agreement with the literature available. The symmetric stretching and bending vibration are assigned the 790 and 450 cm^{-1} respectively are also cited in many literatures.**

Index Terms **- Drying, gel, silicate, spectra, xerogels.**

I.INTRODUCTION

Rare Earth doped glasses are in much demand due to the potential applications in photonics. The Erbium ions in glass matrix exhibit a number of sharp emission lines due to electronic transitions within the 4f shell. The transition from first excited state to the ground state occurs at 1.53 micrometers. Sol-gel process offers an attractive way for preparing rare-earth doped laser materials, optical amplifiers and as fibre amplifiers [1-9]. Samples of various concentrations in Erbium, Ytterbium and Aluminium co-doping had been prepared through the sol-gel route and left unsintered for a period of three years to study for consistency and the change in structural evolution of glass. Several types of phase transformations occur during aging, microsynersis is common in xerogels where solid phase separates from the liquid on a local scale. FT-IR Spectra of the samples exhibited the fundamental vibrations of dry SiO2 near 430 cm-^{1}, 800 cm-^{1}, 1060 cm-^{1} 1200 cm-^{1} [16-22] prominently. In addition to these, defect bands were also observed at 490 cm-^{1} and 600 cm-1 in some of the spectra. The behaviour of the gel during the dehydration and dehydroxylation is conditioned by its microstructure which in turn depends upon the physical conditions prevailing at the time of the hydrolysis. The factors responsible are mixture components, pH of medium, drying conditions.

Microraman Spectra of the samples (Fig. 1) exhibited the fundamental vibrations of dry SiO2 near 430 cm-1, 800 cm-^{1}, 1060 cm-^{1} 1200 cm-^{1} [1] prominently. In addition to these, defect bands were also observed at 490 cm-1 and 600 cm-1 in some of the spectra. The behaviour of the gel during the dehydration and dehydroxylation is conditioned by its microstructure which in turn depends upon the physical conditions prevailing at the time of the hydrolysis. The factors responsible are mixture components, pH of medium, drying conditions. In addition to the maximum intensity, the band also exhibits asymmetricity. The topology of the silicate glasses depends upon the distribution of bond angles and bond distances in the glass. The crystalline materials exhibit halfwidth (Fig. 2 & 3) of less than 5 cm-1 as compared to the halfwidth greater than 50 cm-1 in silicate melts [7]. Thus, halfwidths as a function of variables yield structural information about the silicate sample. These expanded regions appear from thermal agitation and thus a Boltzman type of distribution is expected in silicate gels.

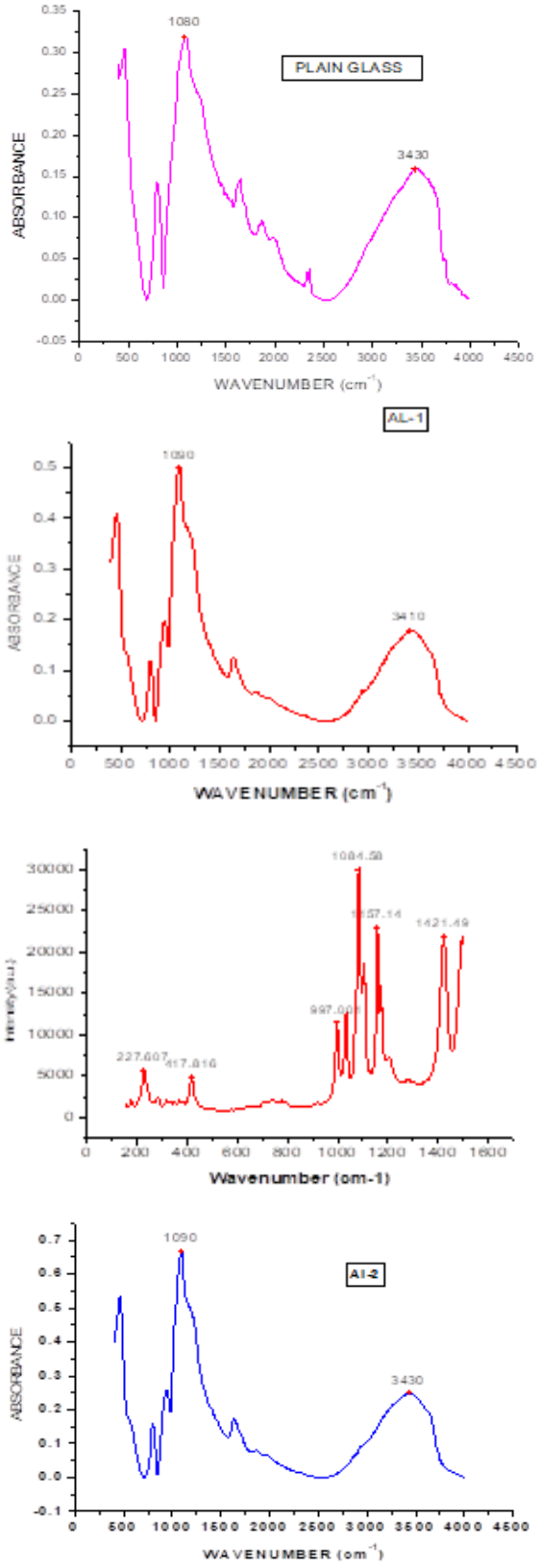

Fig 1: Raman Spectra of Erbium doped xerogel

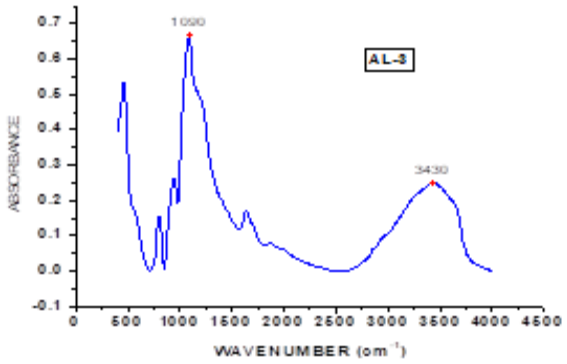

Fig. 2: The FTIR patterns of various concentrations of Er_2O_3 Yb_2O_3 silica gel

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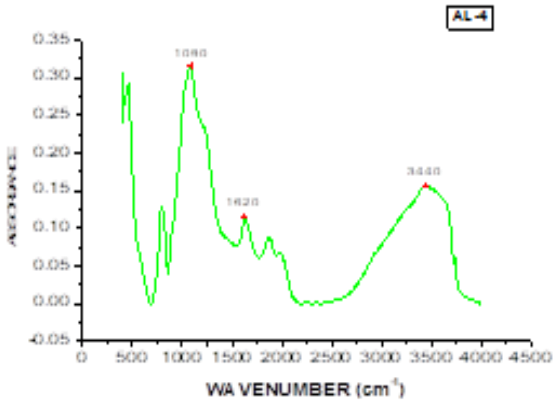

Fig. 3: The FTIR patterns of Er_2O_3 Yb_2O_3 silica gel

II. RESULTS AND DISCUSSIONS

The visual characteristics, the FT-IR data and the EDAX measurements show that the Erbium ions migrate towards the top thereby indicating surface diffusion of do pants towards the upper side on storage. The lower surface has more of dry silica content as supported by FT-IR data at 600 cm^{-1}. The 600 cm^{-1} band diminishes with increase in sintering temperature. Defect structure is assigned to bands around 750 and 550-600 cm^{-1} in agreement with the literature. The symmetric stretching and bending vibration are assigned the 790 and 450 cm^{-1} respectively [1-6]. The peak height of the defect bands is governed by the maximum temperature of sintering (Fig. 4). The effect of increasing the sintering temperature was to lower the peak height at 600 cm^{-1}. The peak at 1100 cm^{-1} exhibited asymmetricity. This indicated a phase separation within the glassy phase due to storage for the period mentioned. An attempt had been made to study the effect of sintering temperature, concentration and nature of do pants. The asymmetric peak is also attributed to superposition of peaks. An attempt is made to isolate the superposed peaks. The relative height of 900 cm^{-1} peak also shows degree of disorder. Raman spectra of the samples are compared with the data in the literature [7-11]. Another interesting feature emerging out of the study indicates a shift of high intensity bands towards higher wavelength in aged samples. Thus, the structure of silicate gels evolved is different from the Spectra of fresh samples.

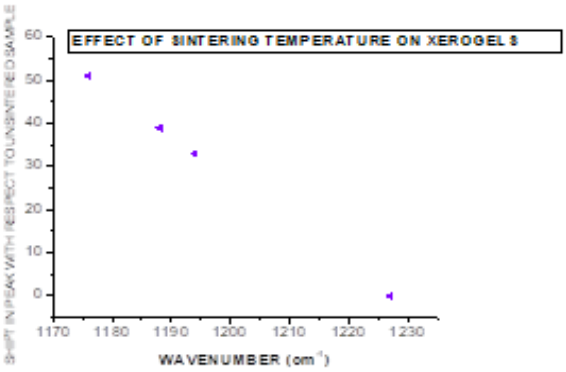

Fig. 4: The effect of various sintering temperatures of Er_2O_3 Yb_2O_3 silica gel

III. CONCLUSIONS

The visual characteristics, the FT-IR data and the EDAX measurements show that the Erbium ions migrate towards the top thereby indicating surface diffusion of do pants towards the upper side on storage. The lower surface has more of dry silica content as supported by FT-IR data at 600 cm^{-1}. The 600 cm^{-1} band diminishes with increase in sintering temperature. Defect structure is assigned to bands around 750 and 550-600 cm^{-1} in agreement with the literature. The symmetric stretching and bending vibration are assigned the 790 and 450 cm^{-1} respectively. The peak at 1100 cm^{-1} exhibited asymmetricity. This indicated a phase separation within the glassy phase due to storage for the period mentioned. The asymmetric peak is also attributed to superposition of peaks. An attempt is made to isolate the superposed peaks. The effect of increasing the sintering temperature was to lower the peak height at 600 cm^{-1}.

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