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EDAX of Aged Silica Xerogels

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Abstract - *Sol-gel glasses were prepared from analytical Grade Tetraethyl Orthosilicate (TEOS), $ErCl_3.6H_2O$, $YbCl_3.6H_2O$, HCl, NH_4OH, de-ionized water and magnetically stirred till a clear liquid formed. The sols were poured into Petri dishes and covered with polythene sheets tied tightly with rubber bands with several pin-holes. They were then kept for seven days at room temperature and then baked in an oven for three weeks at 55°C and were separated into two set of samples. Samples A, were sintered just after baking for three weeks and Samples B, were stored for three years to study the effect of aging, and then sintered.*

Key Words: sintering, erbium, sol-gel, spectral glasses etc.

1. INTRODUCTION

Erbium doped glass has attracted a lot of attention during the last years for its use in integrated optics and compact light sources in eye-safe range (1.55μm spectral range as shown by [1]. In our earlier communication by [2] we reported the synthesis techniques of producing erbium doped glass by the sol-gel route. Transparent and crack free glasses were produced repeatedly. The absorption and emission spectra were studied for suitability as laser materials and Ytterbium concentration was optimized to get the highest absorption coefficient. 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. Erbium doped glass has attracted a lot of attention during the last years for its use in integrated optics and compact light sources in eye-safe range (1.55μm spectral range as shown by [1]. In our earlier communication by [2] we reported the synthesis techniques of producing erbium doped glass by the sol-gel route. Transparent and crack free glasses were produced repeatedly. The absorption and emission spectra were studied for suitability as laser materials and Ytterbium concentration was optimized to get the highest absorption coefficient. 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.

Analytical Grade Tetraethyl Orthosilicate (TEOS), $ErCl_3.6H_2O$, $YbCl_3.6H_2O$, HCl, NH_4OH, de-ionized water were the starting materials for sol-gel glasses. Molar ratios of 1: 14: 0.01 were taken for TEOS: H_2O: HCl and magnetically stirred till a clear liquid formed. At this time, calculated and carefully measured do pants (using Sartorious, single-pan electronic balance) were added and stirred again to mix the salts. Strips were used to check the pH of the solution and NH_4OH was added to yield a pH of 3.5. The sols were poured into Petri dishes and covered with polythene sheets tied tightly with rubber bands with several pin-holes. They were then kept for seven days at room temperature and then baked in an oven for three weeks at 55°C and were separated into two set of samples.

2. EXPERIMENTAL DETAILS

Analytical Grade Tetraethyl Orthosilicate (TEOS), $ErCl_3.6H_2O$, $YbCl_3.6H_2O$, HCl, NH_4OH, de-ionized water were the starting materials for sol-gel glasses. Molar ratios of 1: 14: 0.01 were taken for TEOS: H_2O: HCl and magnetically stirred till a clear liquid formed. At this time, calculated and carefully measured do pants (using Sartorious, single-pan electronic balance) were added and stirred again to mix the salts. Strips were used to check the pH of the solution and NH_4OH was added to yield a pH of 3.5. The sols were poured into Petri dishes and covered with polythene sheets tied tightly with rubber bands with several pin-holes. They were then kept for seven days at room temperature and then baked in an oven for three weeks at 55°C and were separated into two set of samples. Samples A, were sintered just after baking for three weeks and Samples B, were stored for three years to study the effect of aging, and then sintered.XRD was carried out in (20° to 70°) 2θ range using a Philips (PW 1710 diffractometer with CuK_α radiation (λ = 1.5405Å). TGA/DTA analyses were carried out in (Pyris Diamond TGA/DTA analyzer). The FT-IR were measured with the help of (Nicolet, NEXUS 870) Spectrophotometer.

3. RESULTS AND DISCUSSIONS

3.1 Visual Characteristics

The freshly prepared samples were transparent and pink in color. The color deepened with increase in Erbium concentration. The stored samples changed color. The upper (position when allowed to gel from sol) side had brighter pink color after sintering and were almost white (tending towards opacity). This indicated a diffusion of do pants towards the upper side on storage. This is also supported by EDAX measurements before and after storage.

3.2 Structural Characteristics

Chart – 1 shows the XRD patterns of 0.5% Er_2O_3 silica gel sintered at 900°C with a Philips(PW 1710 diffractometer with CuK_α radiation (λ = 1.5405Å). The upper figure was

sintered after three years of gel formation. The graph shows amorphous nature of the sintered gels in glassy phase. There is no crystalline phase up to this sintering temperature before storage or after storage for three years. However sintering to 1100 °C showed distinct peak around 20 °C, as shown in Chart - 1. A: shows the XRD patterns of 0.5% Er_2O_3 silica gel sintered at 900°C for freshly prepared Samples. B: shows the XRD patterns of 0.5% Er_2O_3 silica gel sintered at 1100°C for samples prepared three years back.

3.3 EDAX measurements

The comparison between the above data shows that the stored samples have a massive concentration gradient, probably due to diffusion of Erbium ions in silica gels as reported in visual characteristics.

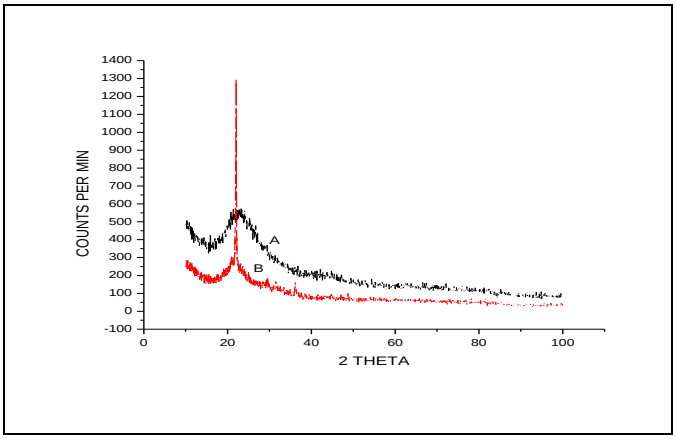

Chart -1: XRD patterns

4. CONCLUSIONS

The visual characteristics, the Micro Raman data and the EDAX measurements show that the Erbium ions migrate towards the top surface and the lower surface has more of dry silica content as shown by Raman data in Table -1.

Table -1: EDAX measurements

EDAX MEASUREMENTS			
Sample	Erbium concentration (%)		
	Top surface	Bottom surface of fresh sample	Bottom surface of aged sample
Fresh	1.5	1.75	1.65
Stored	1.5	1.66	1.14

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