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Influences of Yb^{3+} ion concentration on the spectroscopic properties of silica glass

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We investigated optical spectroscopic properties of silica glass doped with ytterbium (Yb^{3+}) ions to assess their lasing performance in diode-laser pumped systems. The Yb-doped silica glass preforms were fabricated by solution doping technique using the MCVD process. The stimulated emission cross-section and laser performance parameters were determined from the measured absorption spectra using the method of reciprocity. Fluorescence decay characteristics were observed to be deviated from exponential behavior as the Yb^{3+} ions doping level increases and there exists a lifetime quenching behavior related to the cluster effect of Yb^{3+} ions into silica glass. Nevertheless, lasing parameters indicated that clustering of Yb^{3+} ions does not significantly affect the spectral properties relevant to the predicted lasing performance when concentration is low, but becomes predominant at higher concentration.

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1. Introduction

The remarkable advances in fiber laser output powers in recent years [1,2] have led to the search for novel dopants in silica-based fiber with improved glass properties, such as good control of refractive index, better rare-earth (RE) solubility and high optical damage threshold. Nowadays, ytterbium (Yb^{3+}) ion is regarded as the main dopants for the application of high-power diode-pumped laser systems. The long lifetimes (order of 1 ms) exhibited by excited Yb^{3+} ions, allow one to store large energies in the excited state using relatively low or moderate pump powers and this is considered to be an advantage for diode-laser pumping. The Yb^{3+} ion band energy-level scheme is very simple and contains only two multiplet manifolds: the ground $^2F_{7/2}$ and the excited $^2F_{5/2}$. The simplicity of the level structure provides freedom from the unwanted process such as excited state absorption (ESA), multi-phonon non-radiative decay from $^2F_{5/2}$ and obviates the possibility of concentration quenching through cross relaxation. The closeness of the pump and laser wavelengths in combination with features described above contribute to high efficiency (as high as 70% with respect to incident diode-pump power) that can be obtained from Yb^{3+}-doped fiber lasers. In addition, only a small quantum defect exists between the pump and laser wavelengths (∼975 and ∼1030 nm, respectively) and this reduces the thermal load on the host matrix during optical pumping. Silica glass is a very desirable host material for high-power laser applications, because it can be fabricated in the form of a glass fiber and a robust laser design, if Bragg gratings written on the fiber are used as reflectors. On the other hand, silica is a matrix of poor spectroscopic and doping properties inducing clustering, which is extremely harmful and reduces radiative transitions [3,4]. This limitation is generally attributed to the conjunction of RE ions clustering through RE–O–RE bonds with energy transfers between the clustered ions by a cooperative up conversion (CUC) process [5]. So, it is highly desirable to fabricate Yb-doped silica glass for avoiding the RE ions cluster formation caused by the low solubility of rare-earths in silica. Even at lower concentrations, the RE emission properties are subjected to the so-called concentration quenching. In this regard, Al or P-codoping is the one of the proposed solutions to avoid clustering and the consequent concentration quenching [6–9]. Sen et al. suggested that codoping with Al or P results in the formation of structural pockets in the glass consisting of Yb^{3+} ions with oxygen nearest neighbors and Al/P next-nearest neighbors. Such structural pockets give rise to homogenization of RE clusters via a spatial redistribution of the Yb^{3+} ions and an increase in the average Yb–Yb separation distance in the SiO_2 glass structure [9].

Presently our group is carrying out investigations on Yb^{3+} ions incorporation into silica glass preforms using solution doping [10] and chelate [11] methods utilizing modified chemical vapor

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deposition (MCVD) process. The quest for our study is to fabricate high quality Yb-doped silica glass preforms for large mode area-photonic crystal fiber (LMA-PCF) in order to realize high laser output. In relation to this study, we fabricated series of Yb-doped silica glass preforms with or without Al as co-dopant using both techniques. Although, there have been reports on the compositional dependence of the spectroscopic properties of Yb^{3+} ions on various host glass oxides [12–15], investigation regarding Yb^{3+} concentration dependence on the spectroscopic properties of silica glass is relatively scarce. The present paper reports the influences of Yb^{3+} ions concentration on the spectroscopic properties of silica glass fabricated by solution doping technique without using Al as co-dopant. The sole intention for not using Al as co-dopant is to investigate Yb^{3+} ions clustering behavior into silica glass matrix. An attempt is made to explain concentration quenching phenomena via clustering in the Yb-doped silica glasses.

2. Experimental procedure

2.1. Sample preparation

Silica glass based fiber preforms doped with Yb^{3+} ions were fabricated by solution doping technique using the MCVD process. Doping solution of different concentrations was prepared by dissolving ytterbium hexahydrate ($YbCl_3 \cdot 6H_2O$) (Aldrich) into distilled water. Porous core layers of silica glass composition were deposited onto the inside of a silica tube by the MCVD process. Then, the porous layers were soaked with the solution for an hour. After draining the doping solution, the soaked porous layers were dried overnight in N_2 atmosphere, oxidized at ~1100 °C and then sintered at ~1800 °C by flowing helium and oxygen gases. Then the silica glass tube with the sintered core layers was collapsed over 2200 °C to prepare a silica glass fiber preform. Six samples of different concentrations of Yb^{3+} (0.15, 0.20, 0.25, 0.30, 0.60, and 0.85 wt%) were fabricated using the above procedures. The Yb^{3+} concentrations of the core of the preforms were measured by a commercial inductively coupled plasma-mass spectrometry (ICP-MS) technique. Prior to the optical measurements, the sample pieces from the preform were cut to the thickness of 3 mm, and flat polished to optical quality. Absorption spectra were recorded by using a double-beam spectrophotometer (PerkinElmer Lambda 950) in the range 850–1100 nm at room temperature. A Ti-sapphire laser was used to pump Yb^{3+}-doped samples at 930 nm. The power of the incident light was set to be 100 mW on the continuous wave in the 930 nm excitation. The excitation beam was modulated by a mechanical chopper. The $^2F_{5/2} \rightarrow {}^2F_{7/2}$ emission band was measured on a monochromator with an InGaAs diode photomultiplier tube. The fluorescence decay curves of the $^2F_{5/2} \rightarrow {}^2F_{7/2}$ transitions have been measured at room temperature by exciting at 974 nm. Emission lifetimes were obtained from the first e-folding time of decay curves with a digital oscilloscope (Yokogawa, DL-1640). The refractive indices of the samples were measured at different wavelengths by a prism coupler (Metricon, 2010).

2.2. Sample evaluations

2.2.1. Spectral analysis

The absorption cross-section σ_{abs} was calculated by the following equation [15]:

$$\sigma_{abs} = \frac{2.303 \log(I_0/I)}{Nl}, \tag{1}$$

where $\log(I_0/I)$, l, N are the absorbance, sample thickness, Yb^{3+} ions number density (ion/cm^3), respectively.

Exact knowledge of the absorption and emission cross-section spectra is essential for the accurate description and detailed evaluation of the performance of the optical amplifiers and lasers. The two usual methods to obtain the emission cross-section of Yb^{3+}: $^2F_{7/2} \rightarrow {}^2F_{5/2}$ transitions are the reciprocity method [16] and the Fuchtbauer–Ladenburg (F–L) equation [17]. The cross-section obtained by reciprocity method requires the knowledge of the energy diagram of Stark levels of the fundamental and excited states whereas the required parameters for the F–L formula are the emission line shape function, the radiative lifetime and the refractive index. However, due to the presence of radiation trapping process of ytterbium, concentration dependence of fluorescence lifetime and effective width of fluorescence are subject to distortion, and the stimulated emission cross-section calculated by F–L equation is underestimated. For the silica glasses with reasonably high concentration of Yb^{3+} ions, F–L equation is not preferable. On the other hand, the stimulated emission cross-section spectrum obtained according to reciprocity method can be taken as the emission spectra are free from radiation trapping [18,19], and the problem due to the radiation trapping effect in the F–L relation will not occur. In this paper, the emission cross-section of Yb^{3+} ions was calculated by the reciprocity method, by which absolute value of cross-sections and accurate spectral information can be obtained.

The absorption cross-section, σ_{abs}, and the emission cross-section, σ_{emi}, are related according to the following relationship [16]:

$$\sigma_{emi}(\lambda) = \sigma_{abs}(\lambda) \exp\left(\frac{E_{ZL} - hc\lambda^{-1}}{kT}\right), \tag{2}$$

where k is the Boltzmann's constant, E_{ZL} is the zero-line energy which is considered to be the energy separation between the lowest components of the upper and lower states. The zero-line energy, E_{ZL}, is associated with the strong peak in the absorption spectrum of an Yb-doped glass. It can be determined by matching the actual emission spectrum to that of the derived emission result of Eq. (2), since the derived and actual emission spectra agree reasonably well in line shape [17]. Moreover, the absorption line shape is transformed to that of the emission solely on the basis of the $\exp(-h\nu/kT)$ term.

2.2.2. Laser performance parameters

From the absorption and emission cross-section spectra of Yb^{3+}-doped silica glasses, it is possible to obtain some important parameters concerning the laser performance in diode-laser pumped systems. The first important laser parameter, β_{min}, is defined as the minimum fraction of Yb^{3+} ions that must be excited to balance the gain exactly with the ground-state absorption at laser wavelength, λ_l. The quantity of β_{min} is simply given by [12,20]:

$$\beta_{min} = \frac{\sigma_{ab}(\lambda_l)}{\sigma_{ab}(\lambda_l) + \sigma_{emi}(\lambda_l)}, \tag{3}$$

$$= \left\{1 + \exp\left(\frac{E_{ZL} - hc\lambda_l^{-1}}{kT}\right)\right\}^{-1}. \tag{4}$$

When the β_{min} fraction of the Yb^{3+} population is excited, the upward and downward transition rates are equal, and the Yb-doped glass essentially becomes transparent at λ_l, such that there is neither gain nor loss for a weak laser probe beam. Low values of β_{min} are required to have minimal resonant absorption losses at λ_l. Incidentally, $\Delta E = E_{ZL} - hc\lambda_l^{-1}$ neither changes very much in the silica glass, nor does β_{min}.

Pumping dynamics in Yb^{3+} doped glass lasers can be described by the pump saturation intensity, I_{sat}. It is a measure of the ease with which the Yb^{3+} population can be bleached to overcome the ground-state absorption. I_{sat} requires an accurate measure of the

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