Selenide Glass Optical Fiber Doped with Pr^{3+} for U-Band Optical Amplifier

Woon Jin Chung, Hong Seok Seo, Bong Je Park, Joon Tae Ahn, and Yong Gyu Choi

Pr^{3+}-doped selenide glass optical fiber, which guarantees single-mode propagation of above at least 1310 nm, has been successfully fabricated using a Ge-Ga-Sb-Se glass system. Thermal properties such as glass transition temperature and viscosity of the glasses have been analyzed to find optimum conditions for fiber drawing. Attenuation loss incorporating the effects of an electronic band gap transition, Rayleigh scattering, and multiphonon absorption has also been theoretically estimated for the Ge-Ga-Sb-Se fiber. A conventional double crucible technique has been applied to fabricate the selenide fiber. The background loss of the fiber was estimated to be approximately 0.64 dB/m at 1650 nm, which can be considered fairly good. When excited at approximately 1470 nm, Pr^{3+}-doped selenide fiber resulted in amplified spontaneous emission and saturation behavior with increasing pump power in a U-band wavelength range of 1625 to 1675 nm.

Keywords: Selenide, fiber, U-band amplifier, Pr^{3+}.

I. Introduction

To support the rapidly growing demand on the transmission capacity of optical telecommunication networks, co-utilization of wavelength division multiplexing (WDM) and the entire possible transmission window of the silica optical fiber for communication from O-band (1260 to 1360 nm) to U-band (1625 to 1675 nm) may ultimately be necessary. Utilization of the entire window can then be spurred by the installation of OH-free line fiber [1] together with appropriate fiber-optic amplifiers. Fiber amplifiers for O-band [2], S-band [3], and C-band [4], [5] have already been extensively studied and commercialized, while the U-band amplifier has not been greatly explored mainly due to a lack of appropriate material systems in terms of active rare earth (RE) elements and host glasses available for the band. In this regard, it has been a great concern to pursue such an optimized RE dopant and its host glass system for a U-band optical amplifier.

Chalcogenide glasses show very low fundamental vibration energies since they are normally composed with S, Se, or Te acting as a network-forming element including Ge, Ga, As, or Sb as additives. They are transparent up to beyond 10 μm, which renders them great candidates for chemical sensors, mid-infrared light delivery, thermal imaging, and environmental monitoring [6]-[9]. Chalcogenide glass hosts have low phonon energy, which significantly reduces the multiphonon relaxation rates of the doped RE ions compared to conventional oxide glasses [10]. This enables many radiative transitions between two energy levels with a small energy gap and thus a high quantum efficiency of those transitions; this has triggered extensive studies on the mid-infrared laser devices and optical amplifiers based on chalcogenide glasses [7], [8].

Recently, Choi and others observed a 1.6 μm emission from

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the Pr^{3+}: ($^3F_3$, $^3F_4$) → $^3H_4$ transition in selenide glasses, which provided a good opportunity for a new U-band optical amplifier [11], [12]. They characterized the Pr^{3+}-doped Ge-As-Ga-Se bulk glass samples to obtain approximately 212 μs as the lifetime of the excited ($^3F_3$, $^3F_4$) state and approximately 3.1 × 10^{-20} cm^2 as the stimulated emission cross-section of the ($^3F_3$, $^3F_4$) → $^3H_4$ transition. Thus, as a following progress it is crucial to fabricate a low loss selenide fiber for realization of the Pr^{3+}-doped fiber-optic amplifier. Since most of the attenuation loss in usual chalcogenide glasses comes from impurities such as oxides, -SeH, -SH, -OH, and micro-crystallites, fiberization of those glasses needs a well-controlled process minimizing those possible impurities to achieve the theoretical minimum loss [7], [8], [13] and [14]. Sulfide or selenide glass fibers with a transmission loss of a few dB/m in the optical communication window (1.2 to 1.7 μm) have been reported [15]. RE-doped chalcogenide glass fibers have also been fabricated [7], [15] and [16], which were mostly based on As-S (or Se) or Ge-S systems and aimed at their mid-infrared (IR) transitions beyond 2 μm.

In this paper, we prepared Ge-Ga-Sb-Se glasses doped with Pr^{3+} for U-band application, which exhibited comparable spectroscopic properties to the previous system [11], [12]. Ga has been added to improve RE solubility inside the glass network as well as thermal stability against crystallization during the fiberization process. Thermal and optical properties of the bulk glass samples have been investigated to identify suitable drawing criteria and to calculate theoretical minimum attenuation loss of the cylindrical waveguide, which has then been compared to the measured loss of the produced selenide fiber.

II. Experiment

Compositions of $Ge_{30}Ga_2Sb_8Se_{60}$ and $Ge_{30}Ga_2Sb_8Se_{55}S_5$ were used for the core and clad, respectively. Purer than 99.999% praseodymium in its element form doped the core glass. The refractive index of the clad was managed by controlling the amount of S. High purity (> 6N) elemental Ge, Ga, Sb, Se and S were weighed and put into a silica ampoule in a dry box filled with Argon gas. Glasses were melted in a 3-zone rocking furnace at 950°C for 12 hrs and quenched into water followed by annealing at 280°C. Glasses taken out of the ampoule were then cut into a disk shape and optically polished for spectroscopic measurements. A conventional double crucible method was applied to draw selenide fibers with or without a core/clad structure. Fiberization was carried out in a dry box filled with N_2.

A UV/VIS/NIR absorption spectrum was measured using a Perkin-Elmer Lambda 19 spectrophotometer, whereas an FT-IR spectrometer (Model Magna-IR 560, Nicolet) was used for mid-IR transmittance. A differential scanning calorimeter and thermo-mechanical analyzer have been employed to obtain thermal properties such as glass transition temperature (T_g), crystallization temperature (T_x), and viscosity. Spectroscopic properties such as spontaneous emission of the Pr^{3+}: ($^3F_3$, $^3F_4$) → $^3H_4$ transition and its lifetime out of the bulk glass were detected using a photomultiplier tube (PMT, Hamamatsu) when the glass was pumped at 1.48 μm from a high power laser diode. Attenuation loss of the fiber was recorded with an optical spectrum analyzer (Model AQ-63158, Ando) and a white light source (Model TQ8111, Advantest) using a conventional cut-back method within a communication window range of 1100 to 1750 nm.

III. Results

Thermal stability against crystallization is important for fiber drawing of glasses, and (T_x-T_g) is normally used for a measure of the stability. Figure 1 shows the raw data of heat flux variation obtained from differential scanning calorimeter scans for the core ($Ge_{30}Ga_2Sb_8Se_{60}$) and clad ($Ge_{30}Ga_2Sb_8Se_{55}S_5$) glasses when increasing the temperature at 10°C/min. For the core glass, T_g, T_x and (T_x-T_g) were 290°C, 477°C, and 187°C, respectively. The clad glass showed similar values of T_g, T_x and (T_x-T_g), that is, 294, 478, and 184°C, respectively. This indicates that the clad glass modified with S maintains thermal properties of the unmodified core glass. The (T_x-T_g) value of approximately 180 °C represents the high thermal stability of the glasses compared to the conventional chalcogenide glasses [17]. However, it is also important that the glasses should have proper viscosity values for efficient fiber drawings within the temperature window between T_g and T_x. The penetration viscometer method [18] was employed for the measurement of viscosity change with temperature using a conventional thermo-mechanical analyzer. Viscosity (η) at each temperature has been calculated with the modified Nemilov equation (1) suggested by Cardoso and Seddon as follows [18]:

$$\eta = m\frac{Mg}{8\sqrt{A}\dot{\varepsilon}}, \qquad (1)$$

where m represents the geometrical coefficient of the probe (for cylindrical, m = 0.96), M, g and A are the loads applied to the probe, gravity force constant, and area of the probe touching the sample, respectively, and $\dot{\varepsilon}$ is the penetration rate of the probe at a constant temperature. As shown in Fig. 2, the measured data were well fitted with the Vogel-Fulcher equation (represented as a solid line). This clearly shows that the temperature range suitable for fiber drawing (10^5 to 10^7 poise) is 370 to 390°C, which is located in between the thermally

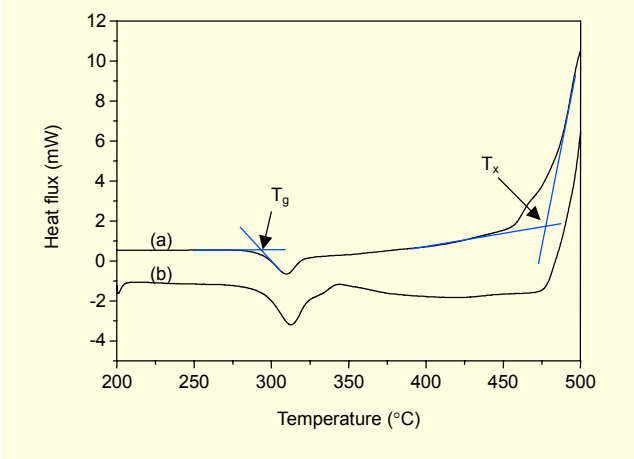

Fig. 1. Differential scanning calorimeter results of (a) the core ($Ge_{30}Ga_2Sb_8Se_{60}$) and (b) clad ($Ge_{30}Ga_2Sb_8Se_{55}S_5$) glasses. The scanning rate was 10 °C/min.

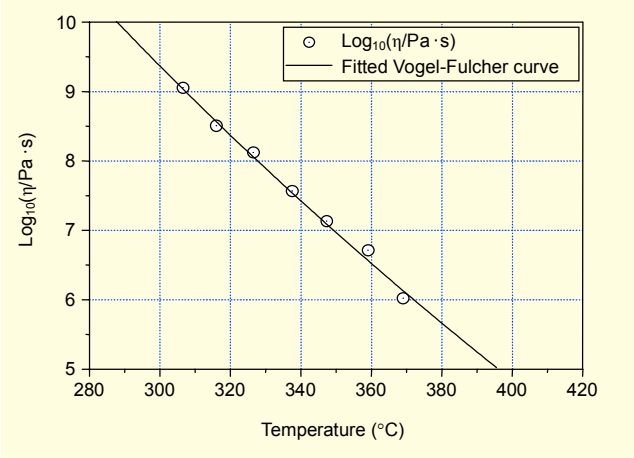

Fig. 2. Viscosity change with temperature measured by penetration viscometer method for $Ge_{30}Ga_2Sb_8Se_{55}S_5$ glass. A solid line represents the fitted result of the data to the Vogel-Fulcher equation.

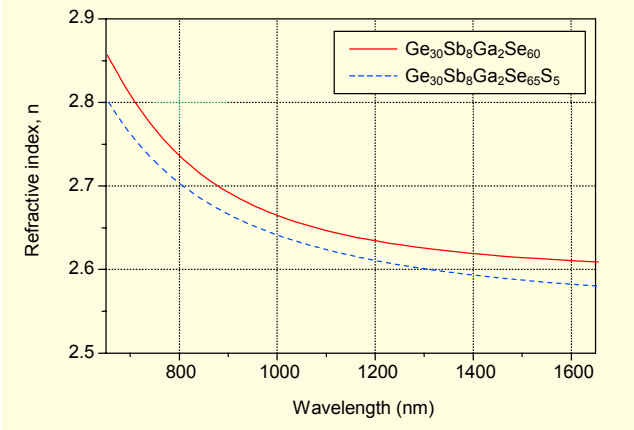

Fig. 3. Refractive index change with wavelengths of the core (solid line) and clad (dashed line) glasses.

stable temperature range (T_g to T_x). This implies that the present system would show efficient fiber drawing properties along with the well-matched T_g and T_x of the core and clad glasses.

Optical properties of the bulk glasses were also inspected. A UV/VIS/NIR absorption spectrum and FT-IR spectrum of the core glass were measured. The UV/VIS/NIR revealed the optical band gap of the glass as approximately 1.47 eV, which is small compared to other glasses such as sulfides, fluorides, or oxides glasses. Transmittance within the 400 to 4,000 cm^{-1} range indicated almost no contamination induced by -OH, -SeH, -GeH or CO_2. A refractive index change of the glasses against the wavelength was obtained using an ellipsometer and is shown in Fig. 3. It has been reported that 1 at % substitution of Se with S in As-Ge-Se glasses reduces the refractive index by approximately 0.005 at 1.8 μm [19]. A similar effect was observed in the clad glass by replacing 5 mol% of Se with S. However, the effect of S introduced in Ge-Sb-Se glasses was larger than that in As-Ge-Se glasses. This can possibly be attributed to the difference in modifier ions (Sb or As) or dielectric constant fluctuation induced by the S in each glass. Emission properties of a 0.02 mol% Pr^{3+}-doped core glass are depicted in Fig. 4. When pumped with a 1.47 μm laser diode, it features a characteristic emission spectrum from the Pr^{3+}: ($^3F_3$, $^3F_4$) → $^3H_4$ transition centered at a 1.65 μm well-covering U-band (1625 to 1675 nm). The inset of Fig. 4 represents the population decay profiles of the ($^3F_3$, $^3F_4$) level with the fluorescence lifetime of approximately 270 μs. The well-fitted single exponential decay of the fluorescence indicates almost pure radiative transition characteristics of the decay. Choi and

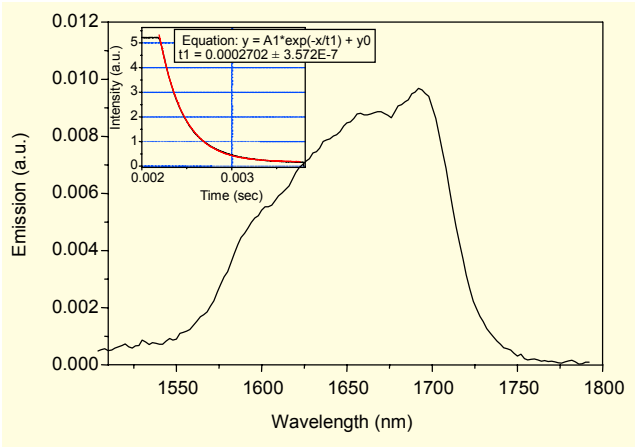

Fig. 4. Emission spectrum of the 0.02 mol% Pr^{3+} doped core ($Ge_{30}Ga_2Sb_8Se_{60}$) glass when pumped with an approximately 1.47 μm laser diode. The Inset figure represents both the fluorescence decay curve of the Pr^{3+}:$^3F_{3,4}$ level when monitored at 1.65 μm in wavelength, and its fitted result to the single exponential decay function (dotted line).

others [11] reported a similar lifetime (approximately 212 μs) in Ge-As-Sb-Se glasses and the highest product of $\sigma_{se}\tau_m$ for a U-band compared to sulfide and fluoride glasses, where σ_{se} and τ_m represent the stimulated emission cross section and the measured lifetime of the excited energy level, respectively.

Ge-Sb-Se glasses showed crystallization during the following heat treatment process for preform fabrication when they were doped with Pr^{3+}. With the addition of Ga, the crystallization of the glass could be avoided due to the improved solubility of the rare earth ions in the glass network. With a good thermal stability for fiber drawing and similar spectral property of Pr^{3+} to that of Ge-As-Sb-Se glasses, Ge-Ga-Sb-Se glasses are expected to be a strong candidate for a U-band optical fiber amplifier.

IV. Discussions

Theoretical attenuation loss of a glass predicts a minimum loss of the fiber, which can be achieved using the same composition as the glass. Thus, it is important to calculate the theoretical loss of the fiber to evaluate the quality of the fabricated fiber and expect the minimum loss of the fiber within the whole transmission window of the glass. Absorption induced by the electronic transition, Rayleigh scattering, and the multiphonon absorption are responsible for the attenuation of light passing through a medium [20]. Attenuation loss due to the electronic transition (α_e) as well as the multiphonon absorption (α_{mp}) was obtained by fitting (2) to the electronic (or multiphonon) absorption edge in a UV/VIS/NIR spectrum or FT-IR spectrum as described previously [21], [22].

$$\alpha = A\exp(B\nu), \qquad (2)$$

where A and B are the host-dependent parameters, while ν is the wave number in cm^{-1}. Rayleigh scattering loss (α_R) was calculated considering the density fluctuations and compositional fluctuations within the glass structure using various material parameters of the present glass following the process previously reported [20]-[22]. Figure 5 displays the resultant theoretical attenuation loss of the fiber. As found in the figure, the minimum loss of the Ge-Ga-Sb-Se fiber is expected to be about 3.9×10^{-4} dB/m at approximately 6.4 μm (approximately 1560 cm^{-1}). The minimum achievable background loss at 1.65 μm (about 6060 cm^{-1}) is predicted to be approximately 0.4 dB/m. It should be noted that the attenuation loss due to the electronic absorption is greatly influenced by the weak absorption tail in a glass with low optical band gap such as selenide glasses [22]. Thus, the electronic attenuation loss should be confirmed with the loss obtained by a sufficiently thick sample. However, the present glass showed almost no change in its loss varying sample thickness from 1 to 20 mm. This implies that there exists few inter-band gap states such as defects or impurities contributing to the weak absorption tail in a Ge-Ga-Sb-Se glass network [22], [23].

Fibers with core/clad structure have been fabricated using a conventional double crucible method [24]. The ratio between the core and clad as well as the fiber thickness have been controlled by the Ar pressure, operation temperature, and pulling speed of the fiber. An uncoated and structureless fiber has also been produced to measure the attenuation loss of the fiber minimizing any external scattering sources. The nominal diameter of the fiber was around 110 μm. Transmission of the structured fibers has been inspected using a tunable laser source after butt coupling of the selenide fiber with a single mode silica fiber. Figure 6 represents transmission images of the fibers with different core diameter when a 1.31 μm laser source was launched to avoid possible ground state absorption of the Pr^{3+}: $^3H_4 \rightarrow (^3F_3, {}^3F_4)$ transition. A near field image corresponding to only single mode propagation after approximately 30 cm has been clearly identified as shown in Fig. 6(a), while propagation of higher modes are shown in Fig. 6(b). The cutoff wavelength of the second lowest order mode has not been confirmed due to the high loss of the fiber at a shorter wavelength than 1.31 μm, which is caused by the Urbach tail absorption of the electronic transition in selenide glasses.

The transmission loss of the fiber has been measured using a white light source equipped with optical spectrum analyzer. A conventional cut-back method has been employed for both fibers with and without a core/clad structure. Measured transmission loss for structureless fiber doped with 0.02 mol%

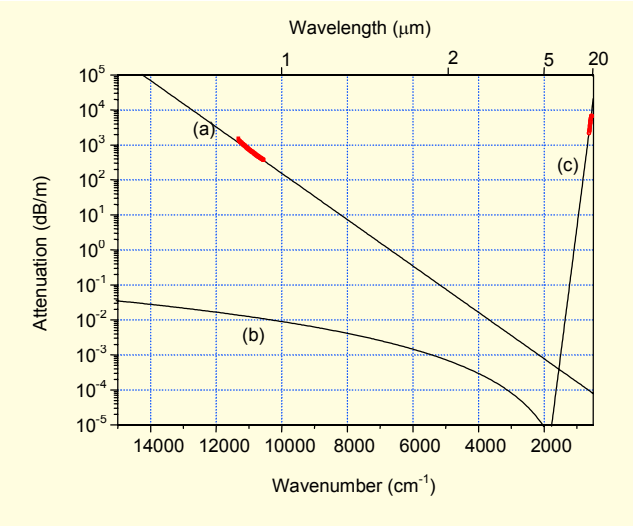

Fig. 5. Theoretically calculated attenuation loss based on the experimental results for $Ge_{30}Ga_2Sb_8Se_{60}$ glass considering (a) electronic transition, (b) Rayleigh scattering, and (c) multiphonon absorption within the glass.

of Pr^{3+} is shown in Fig. 7 as a solid line. Absorption due to the Pr^{3+}: $^3H_4 \rightarrow (^3F_3, ^3F_4)$ transition was clearly observed in a wide range between 1.4 and 1.7 μm. The background loss of the fiber was fitted to the same exponential relationship as (2) assuming the dominant effect of the electronic transitions in this energy region. The result is depicted as a dashed line which fitted well with the experimental data. It predicts that the background loss of the fiber is approximately 0.64 dB/m at 1.65 μm which is similar to the calculated minimum achievable attenuation loss (about 0.4 dB/m) in Fig. 5. The measured loss also represented approximately 0.7 dB/m at around 1.75 μm including the absorption tail of the Pr^{3+}: $^3H_4 \rightarrow (^3F_3, ^3F_4)$ transition. This is a highly improved result compared to the fiber drawn from Ge-As-Ga-Se glasses doped with Pr^{3+}, which showed > 4 dB/m at approximately 1.75 μm [25], or fiber with a similar composition of Ge-Ga-Sb-Se [26].

Fig. 6. Transmitted image of the 1.31 μm light source at the end of the fabricated selenide fiber using (a) single mode and (b) multi-mode. The image was taken from a CRT monitor connected with an IR CCD camera.

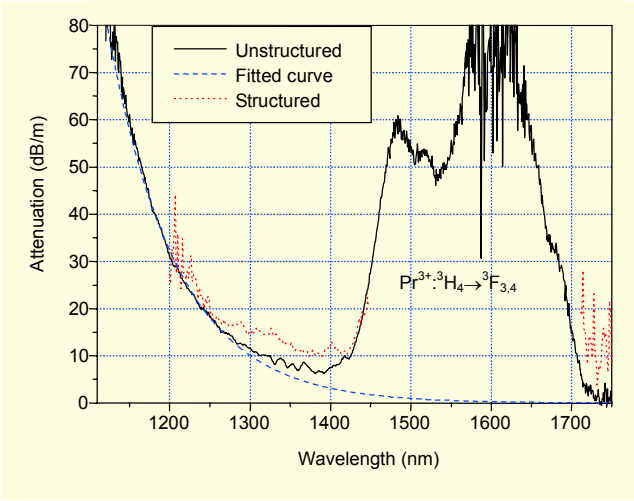

Fig. 7. Measured attenuation loss of the $Ge_{30}Ga_2Sb_8Se_{60}$ glass fiber doped with 0.02 mol% Pr^{3+} without cladding material (solid line) and its fitted result for the background loss (dashed line). Attenuation loss measured from the multi-mode fiber is also presented (dotted line).

This suggests that the fabricated selenide fiber in this study maintains high quality in its transmission loss and has high potential for possible applications in both active and passive optical devices. However, as represented as a dotted line in Fig. 7, the structured fiber in multimode shows a higher transmission loss than the structureless fiber. Scattering loss at the interface between the core and clad could be responsible for the deteriorated loss along with the possible incorporation of impurities both in the core and interface. Decent control of the materials as well as the core/clad interface is required for further improvement of the transmission loss.

A 1.49 μm laser diode has been coupled to the 0.05 mol% Pr^{3+}-doped multimode fiber (about 30 cm) to investigate the spontaneous emission characteristics. Spontaneous emission due to the transition from the thermally coupled Pr^{3+}: $(^3F_3, ^3F_4)$ energy level to the ground state ($^3H_4$) has been observed. Further increase of the pump power can build up the population of the excited state and will generate the amplified spontaneous emission spectrum similar to the bulk samples with proper management of the fiber length. The possibility of population inversion of the Pr^{3+}-ion, which makes the fiber yield a gain in the U-band, has been tested with the peak intensity change at 1.64 μm varying pump power, as shown in Fig. 8. Peak intensity normally shows saturation after population inversion has been achieved. It can also be observed in Fig. 8, suggesting that the U-band optical amplifier can be attained with the present low-loss selenide fiber by proper management of the fiber length and pump power. However, relatively high pump power seems to be necessary for the full inversion of the Pr^{3+} ions in selenide fiber. High coupling loss at the input surface of the selenide glass due to its high refractive index could be responsible for the phenomena.

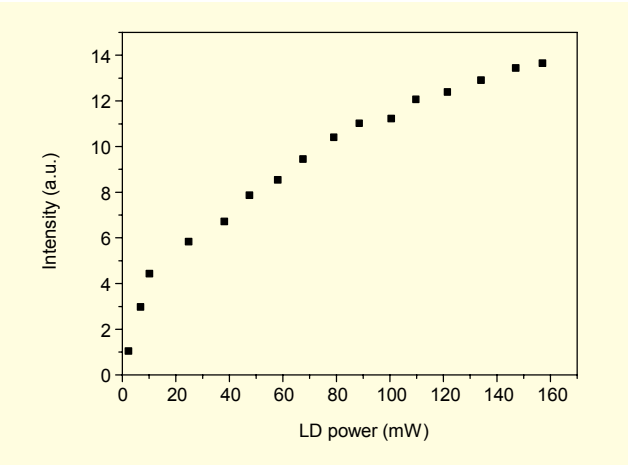

Fig. 8. Peak intensity change of the spontaneous emission varying output power of the 1.49 μm laser diode. Emission intensity has been monitored at 1.64 μm with OSA resolution of 10 nm.

Transition of the electron from the excited state (Pr^{3+}:$^3F_3$, $^3F_4$) to the higher states of the Pr^{3+} ion or into the conduction band of the glass can also consume the pump energy, both of which may disturb the accumulation of population at the Pr^{3+} : ($^3F_3$, $^3F_4$) level. As pump power increases, a nonlinear effect such as two-photon absorption can be activated due to the high nonlinearity of the host matrix, which may further spend pump energy. These processes are likely to happen in glasses with small optical band gap energy such as selenide glasses. Increasing the band gap of the host matrix via compositional change or introduction of the sensitizer such as Er^{3+}-ion [11], [12] in the host glass should be considered to minimize pump power loss within the core.

V. Conclusion

A good fiber drawing ability of the Ge-Ga-Sb-Se host glasses has been confirmed from their thermal stability against crystallization and viscosity variation with temperature. A refractive index change by the introduction of 5 mol% S in clad glass has been monitored at varying wavelengths. A U-band emission of the Pr^{3+} centered at 1.65 μm has also been observed with a reasonable lifetime of approximately 270 μs. The minimum achievable background loss of the fiber at 1.65 μm has been expected to be about 0.4 dB/m by calculation of the theoretical attenuation loss taking into account electronic transition, Rayleigh scattering, and multiphonon absorption of the glass. Based on these thermal and optical properties, a series of Pr^{3+}-doped selenide fiber has been successfully fabricated through the conventional double crucible method. Background loss of the structureless fiber has been measured as approximately 0.64 dB/m at 1.65 μm, which was comparable to the minimum achievable attenuation loss of the fiber. Single mode transmission at 1.31 μm has been confirmed in our fiber with a core/clad structure. Spontaneous emission of the Pr^{3+} at the U-band has been detected with its possibility of population inversion to yield an optical gain. These results exhibited high potential of the Ge-Ga-Sb-Se glass as a host material for a U-band fiber-optic amplifier. Further study is required to minimize the attenuation loss of the structured fiber and pump energy loss inside the RE-doped core.

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