Raman scattering enhancement characteristic of NbCl₅- and Nb₂O₅-doped silica fibers

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Received March 25, 2016; accepted August 12, 2016; posted online September 19, 2016

Two kinds of Nb-doped silica fibers, an NbCl₅-doped fiber and an Nb₂O₅-doped fiber, are fabricated and characterized in this Letter. First, the refractive index profiles of both fibers are obtained, and then their Raman spectra are measured with 785 nm exciting light. The Nb-doped fibers' Raman spectra are compared with a conventional GeO₂-doped single-mode silica fiber that is prepared with the same method and under the same conditions. As a result, the Raman gain coefficients of the Nb-doped silica fiber core are obtained. The experimental results show that Nb₂O₅ doping can enhance the Raman scattering intensity of the optical fibers.

OCIS codes: 060.2270, 290.5860, 060.2400, 060.2290.

doi: 10.3788/COL201614.100602.

Stimulated Raman scattering (SRS) is a very important non-linear effect in optical fibers. In optical fiber communication, SRS can cause an energy transfer between adjacent channels, which limits the multichannel fiber optic system's performance. However, people still take advantage of SRS in fibers, and its excellent performance can be used in broadband Raman amplifiers, Raman fiber lasers, and Raman fiber sensors [1-5]. In these applications, we expect to have optical fibers that have a high Raman gain but low loss to be applied as a Raman amplifying medium. At present, researchers have studied many kinds of Raman scattering-enhanced materials, which can be made of new optical fibers, such as thallium-tellurium oxide glass, a single-crystal sapphire fiber, and a Yb:YAG crystal-derived optical fiber $\frac{[6-9]}{2}$. Among the advanced materials for Raman amplification, one of the most interesting is oxide glass. This is because the optical properties of oxide glasses can be easily modulated, acting on both the chemical composition and the microstructure. Thallium-tellurium oxide glasses and chalcogenide-based glasses combine a large scattering intensity and bandwidth, giving very high Raman gains as compared to pure SiO_2 . As₂Se₃ and As₂S₃ glasses also attracted particular interest, and researchers have studied many characteristics of the glass and the fiber^[10]. It is known from the spectra that spontaneous Raman scattering of these glasses are much more intense than those of fused silica. This property has stimulated interest in other Raman gain media, and multiple glasses have been investigated, usually after being drawn into fibers. Although the glasses will ultimately be used in fiber form, the fabrication of experimental fibers from new glasses is a complex and time-consuming process. So their fabrication demands particularly careful precautions, including a controlled melting atmosphere. Thus, silicon dioxide-based glasses,

due to their compatibility with existing optical fiber technology and the low loss, are more attractive than tellurite-like materials. In these works, Nb_2O_5 can be found doped in tellurite classes and silicon dioxide-based glasses, and it can enhance the Raman gain characteristic of the glasses^[11,12].

In this Letter, novel specialty fiber, Nb-doped silica fibers were fabricated with the modified chemical vapor deposition method. Meanwhile, a standard single-mode optical fiber, which was a GeO₂-doped silica fiber without the Nb dopant, was also made as the contrast of the Nb-doped fiber with the same technology. The Raman spectra of the NbCl₅-doped fiber and the Nb₂O₅-doped fiber were measured and compared with the standard single-mode optical fiber.

We measured the refractive index profiles of the NbCl₅-doped fiber and Nb₂O₅-doped the fiber with fiber index analyzer S14, and the results are shown in Figs. <u>1</u> and <u>2</u>, respectively. The refractive index difference between the fiber core and cladding of the NbCl₅-doped fiber is about 0.0095. As for the Nb₂O₅-doped fiber, it is a little higher, around 0.0135.



Fig. 1. Refractive index profile of $NbCl_5$ -doped fiber.



Fig. 2. Refractive index profile of Nb_2O_5 -doped fiber.

Optical amplification via SRS, called Raman amplification, has the advantages of self-phase matching between the pump and the signal, and a broad bandwidth or high-speed response, compared with other non-linear processes.

When a weak signal is launched with a stronger pump, it will be amplified due to SRS. The amplification of the signal is described through the following equations^[13]:

$$P_S(L) = P_S(0) \exp\left(\frac{g_R(\nu)P_0L_{\text{eff}}}{KA_{\text{eff}}} - \alpha_S L\right), \qquad (1)$$

$$g_R(\nu) = \sigma_0(\nu) \cdot \frac{\lambda_S^3}{c^2 h n(\nu)^2}, \qquad (2)$$

where c is the velocity of light, h is Planck's constant, λ_s is the Stokes wavelength, $n(\nu)$ is the refractive index, which is frequency dependent, and K is the polarization factor, which is 1 if the pump and signal waves are polarization matched, or 2 if they are depolarized. The spontaneous zero Kelvin Raman cross section $\sigma_0(\nu)$ is defined as the ratio of the radiated power at the Stokes wavelengths to the pump power at temperature 0 K.

In the experiments, we measured the Raman spectra of the NbCl₅-doped and Nb₂O₅-doped silica fiber preforms, the Nb₂O₅-doped and Nb₂O₅-doped silica fibers, and the contrast GeO₂-doped silica fiber. The schematic of the experiment is shown in Fig. 3. The system mainly includes a 785 nm laser, a Raman spectrometer, and a computer control system. Through a microscope, the 785 nm laser is coupled into the fiber, and the spontaneous backward Raman scattering light in the fiber is collected into the spectrometer by the same microscope. The Raman spectrum reflects the molecular vibration mode, and the Raman gain spectra of different wavelengths of exciting light have no significant difference. But the Raman gain spectrum measurement of the doped fiber is susceptible to be interfered with by the fiber's fluorescence characteristic, so a 785 nm laser is used as the pump source in the experiment, which could effectively avoid the interference of the fluorescence effect in the fiber and ensures the accuracy of the optical fiber's Raman gain spectrum measurements.

The Raman gain coefficient is an important parameter in measuring the Raman gain properties of a material.



Fig. 3. Raman spectrum measurement apparatus for Nb-doped optical fiber.

Methods of measuring the Raman gain coefficient of materials have been reported^[14,15]. One method used to obtain the Raman gain coefficient is based on the spontaneous Raman spectra. This method needs to measure the spontaneous Raman scattering spectra of the test sample and the standard sample (typically SiO₂) under the same conditions and then uses normalization to obtain the test sample's Raman gain coefficient.

With this method, we measured the Raman spectra of the fiber preform slices' cladding section (pure silica) and the Nb-doped core under the same conditions, and then we used normalization to obtain the Raman gain coefficient of the Nb-doped silica core. The results are shown in Fig. 4. The Raman gain coefficient of pure SiO₂ is $1.26 \times$ 10^{-13} m/W at a frequency shift of 440 cm⁻¹ for 785 nm excitation. This value was obtained by simply scaling the experimental gain coefficient of silica for 532 nm pumping, assuming $1/\lambda_{\text{pump}}$ dependence^[16]. It can be obtained from Fig. 4 that the Raman gain coefficient of the silica core doped with NbCl₅ is about 1.83×10^{-13} m/W, and the other one doped with Nb₂O₅ is 2.68×10^{-13} m/W at 440 cm^{-1} for 785 nm pumping. Therefore, the Raman gain coefficients of the silica cores doped with niobium and germanium were significantly improved.

To better analyze the Nb-doped fiber's Raman gain characteristics, the Raman spectra of the NbCl₅-doped



Fig. 4. Raman gain coefficient of the Nb-doped silica core of the fiber preform slice.

fiber, Nb₂O₅-doped fiber, and contrast GeO₂-doped fiber are all measured under the same conditions. In the experiments, the length of the fibers under test is 10 m and the power of the 785 nm exciting light is 15 mW. The measured Raman gain spectra of the NbCl₅-doped fiber and standard fiber are shown in Fig. <u>5</u>.

As can be seen from Fig. 5, the main Raman peaks of the NbCl₅-doped silica fiber and the standard fiber are almost at the same location. In the vicinity of the frequency shift band at 445 cm^{-1} is symmetrical stretching of the bridging oxygens related to six membered SiO_4 rings: Si-O-Si. The wave numbers of 491 and 605 $\rm cm^{-1}$ are also assigned to the breathing motions of the bridging oxygens, and the shift at 491 cm⁻¹ is associated with 4 membered SiO_4 rings, while 605 cm^{-1} is similar to 3 membered SiO₄ rings. The asymmetric broadband at around 800 cm^{-1} is the TO/LO split bending modes. In the 1060 $\rm cm^{-1}$ peak, there is obvious asymmetrical stretching of bridging oxygens. Although the two fibers have the same Raman shifts, when their Raman intensities are compared, the NbCl₅-doped fiber is higher than the single-mode fiber at 445 cm^{-1} . From the Raman gain spectrum in Fig. 5, one can see that for the Raman gain intensity at the Raman shift 960 cm^{-1} , the standard fiber is the same as the NbCl₅-doped fiber. But for the silica fiber, we are more concerned about the main Raman gain band at 445 cm^{-1} . Thus, in some applications, such as Raman fiber amplifiers, when using an optical fiber as the Raman gain medium, the same length of NbCl₅-doped specialty fiber will have a higher Raman gain than an ordinary single-mode silica fiber for the Raman fiber amplifier.

Figure <u>6</u> shows the results of the Nb₂O₅-doped fiber compared with the standard fiber. It indicates that the Raman scattering intensity of the Nb₂O₅-doped silica fiber is significantly more enhanced than the standard fiber, especially at the wavenumber 445 cm⁻¹. Comparing the measurement results shown in Figs. <u>5</u> and <u>6</u>, it can be found that the Raman peak intensity of the NbCl₅-doped fiber is approximately 1.5 times more than that of the standard fiber at 445 cm⁻¹, and the Nb₂O₅-doped fiber is about 3.5 times more than the standard fiber.

From the experimental results, it can be seen that the Raman scattering intensities of both the $NbCl_5$ -doped and



Fig. 5. Raman spectra of $\rm NbCl_5\text{-}doped$ optical fiber and conventional GeO_2-doped single-mode fiber.



Fig. 6. Raman spectra of $\rm Nb_2O_5\text{-}doped$ optical fiber and conventional GeO_2-doped single-mode fiber.

the Nb₂O₅-doped silica fibers are significantly enhanced more than the ordinary single-mode silica fiber. In particular, for the Nb₂O₅-doped silica fiber, the Raman scattering intensity is about 3.5 times more than the standard singlemode optical fiber. Different materials doped in the core of a silica fiber will change the non-linear optical property of the optical fiber. Because the molecular structure and energy level structure of the fiber materials are different, the Raman gain of the fiber may have a certain amount of enhanced features.

In order to further analyze the contribution of the NbCl₅ and Nb₂O₅ dopants to the Raman enhancement, GeO₂'s contribution needs to be excluded. Based on the measured refractive index profile of the Nb-doped fibers in Figs. <u>1</u> and <u>2</u>, we can obtain the GeO₂-doping concentration of both fibers^[17]. The change in the Raman cross section with GeO₂-doping mol% (χ_{GeO_2}) in pure silica has been summarized in^[18]:

$$\sigma_0'(\chi_{\text{GeO}_2},\nu) = \sigma_0'(\text{SiO}_2,\nu) + C(\nu) \cdot \chi_{\text{GeO}_2}, \qquad (3)$$

where $\sigma'_0(\text{SiO}_2, \nu)$ and $\sigma'_0(\chi_{\text{GeO}_2}, \nu)$, respectively, represent the Raman cross sections of pure silica and GeO₂-doped silica, normalized with respect to the peak Raman cross section of pure silica, and $C(\nu)$ is a linear regression factor (χ_{GeO_2}) whose value at different frequency shifts $\nu^{[18]}$.

In the fiber core of the NbCl₅ and Nb₂O₅ fibers, GeO_2 and compounds containing niobium coexist. Then, the Eq. (3) can be written as

$$\begin{aligned} \sigma_0'(\chi_{\text{GeO}_2,\text{Nb}},\nu) &= \sigma_0'(\text{SiO}_2,\nu) + C_{\text{GeO}_2}(\nu) \cdot \chi_{\text{GeO}_2} \\ &+ C_{\text{Nb}}(\nu) \cdot \chi_{\text{Nb}}. \end{aligned}$$
(4)

From the refractive index difference between Figs. $\underline{1}$ and $\underline{2}$, we obtained the mole percentage of GeO₂ in NbCl₅ and Nb₂O₅ fibers as 7.49 and 9.21, respectively. Then, $C_{\text{GeO}_2}(\nu) \cdot \chi_{\text{GeO}_2}$ can be obtained for the NbCl₅ and Nb₂O₅ fibers. By comparing these with the experiment results in Fig. $\underline{4}$, the calculated results of $C_{\text{Nb}}(\nu) \cdot \chi_{\text{Nb}}$ are shown in Fig. $\underline{7}$.

Taking into account the experimental error, it can be seen from Fig. 7 that the linear regression factor of NbCl₅ is around zero. This means that the NbCl₅ dopant



Fig. 7. Linear regression factor.

makes little contribution to the fiber's Raman scattering enhancement. But for Nb₂O₅, $C_{\rm Nb}(\nu) \cdot \chi_{\rm Nb}$ is about 0.5 during the frequency shift from 350 to 470 cm⁻¹. Therefore, the Nb₂O₅ dopant actually enhanced the Raman scattering intensity of the fiber.

Nb-doped specialty fibers are prepared and investigated with specific reference to the Raman gain spectrum. A significant Raman gain enhancement with respect to a standard single-mode optical fiber is demonstrated. From the application point of view, the merits of our results are related to the advantages of the investigated Nb-doped optical silica fibers, which demand no challenging fabrication and are also compatible with the current optical fiber technology. Additionally, Nb₂O₅-doped optical silica fibers have a higher Raman gain than standard single-mode fibers but have a lower loss than metal-oxide glass optical fibers. This shows us that by selecting a specific doping material such as Nb_2O_5 and using the appropriate process incorporate it into a normal silica fiber, it will be possible to obtain a specialty fiber with Raman-enhancing properties, which is valuable for future applications of non-linear optical devices.

This work was supported by the National Natural Science Foundation of China (Nos. 61027015, 61177088, 61475095, and 61575120), the National "973" Program

of China (No. 2012CB723405), and the Key Laboratory of Specialty Fiber Optics and Optical Access Networks (Nos. SKLSFO2012-01, SKLSFO2013-02, and SKLSFO2015-01).

References

- Y. Ke, B. Zhang, W. Yang, H. Chen, and J. Hou, Opt. Express 21, 14272 (2013).
- M. Hu, W. Ke, Y. Yang, M. Lei, K. Liu, X. Chen, C. Zhao, Y. Qi, B. He, X. Wang, and J. Zhou, Chin. Opt. Lett. 14, 011901 (2016).
- X. Du, H. Zhang, X. Wang, P. Zhou, and Z. Liu, Photon. Res. 3, 28 (2015).
- W. Wang, L. Huang, J. Leng, S. Guo, and Z. Jiang, Chin. Opt. Lett. 12, S21401 (2014).
- Z. Zhang, J. Wang, Y. Li, H. Gong, X. Yu, H. Liu, Y. Jin, J. Kang, C. Li, W. Zhang, W. Zhang, X. Niu, Z. Sun, C. Zhao, X. Dong, and X. Jin, Photon. Sens. 2, 127 (2012).
- M. Irannejad, G. Jose, A. Jha, and P. Steenson, Opt. Commun. 285, 2646 (2012).
- N. Manikandan, A. Ryasnyanskiy, and J. Toulouse, J. Non-Cryst. Solids 358, 947 (2012).
- C. Raml, X. N. He, M. Han, and D. R. Alexander, Opt. Lett. 36, 1287 (2011).
- 9. P. D. Dragic and J. Ballato, Electron. Lett. 49, 895 (2013).
- X. Gai, D. Y. Choi, and S. Madden, J. Opt. Soc. Am. B 28, 2777 (2011).
- Y. Guo, A. Schulte, R. Stegeman, G. Stegeman, C. Rivero, K. Richardson, and T. Cardinal, in *Conference on Lasers and Electro-Optics. Technical Digest (CD)* (Optical Society of America, 2005), paper CME2.
- R. Stegeman, C. Rivero, and K. Richardson, Opt. Express 13, 1144 (2005).
- R. H. Stolen, S. E. Miller, and A. G. Chynoweth, Nonlinear Properties of Optical Fibers, Optical Fiber Telecommunications (Academic, 1979).
- M. D. O'Donnell, K. Richardson, and R. Stolen, Opt. Mater. 30, 946 (2008).
- R. Stegeman, C. Rivero, and G. Stegeman, J. Opt. Soc. Am. B 22, 1861 (2005).
- 16. R. H. Stolen and Clinton Lee, J. Opt. Soc. Am. B 1, 652 (1984).
- Y. Kang, Calculations and Measurements of Raman Gain Coefficients of Different Fiber Types (Virginia Tech, 2002).
- S. T. Davey, D. L. Williams, B. J. Ainslie, W. J. M. Rothwell, and B. Wakefield, IEE Proc. J. Optoelectron. 136, 301 (1989).