Investigation of \(Y_{2.1}\text{Er}_{0.9}(\text{Sc}_{x}\text{Ga}_{1-x})_5\text{O}_{12}\) Matrix Components on the Spectral Properties around 3.0 \(\mu\text{m}\) by Micro-Pulling-Down Method

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Abstract: Single crystal fibers of 30% \(\text{Er}^{3+}\)-doped compound of \(Y_3(\text{Sc}_x\text{Ga}_{1-x})_5\text{O}_{12}\) have been grown by using the micro-pulling down (\(\mu\text{-PD}\)) technique successfully. Our main purpose is to tune the fluorescence properties by adjusting the ratios of \(\text{Sc}^{3+}\) and \(\text{Ga}^{3+}\) ions inside the matrix crystals. The crystal structures of the series compounds were measured and analyzed through X-ray diffraction (XRD) measurements. The components and doping elements distributions were measured by the X-ray Fluorescence spectrometry and electron-probe microanalyzer. The absorption and mid-infrared fluorescence spectra, including the fluorescent lifetime of \(\text{Er}^{3+}: 4\text{I}_{13/2}\) and \(4\text{I}_{11/2}\) levels were measured and compared systematically at room temperature. Spectral analysis indicated that the fluorescent lifetime of \(\text{Er}^{3+}: 4\text{I}_{13/2}\) tended to shorten and the emission spectra began to show a red shift when the proportion of YSG increased in the compound. Furthermore, the Raman spectra were measured to reveal the variations of lattice vibration and phonon energy.

Keywords: \(Y_3(\text{Sc}_x\text{Ga}_{1-x})_5\text{O}_{12}\) crystal; mid-infrared fluorescence spectra; \(\mu\text{-PD}\)

1. Introduction

The laser in the region of 2.7–3.0 \(\mu\text{m}\) has attracted much attention because of the wide applications of communications, medicine, remote sensing and science research [1–4]. The laser shows a weak absorption of water at the wavelength of 2.911 \(\mu\text{m}\). As a result, it can lose little energy when going through the atmosphere to the space. The Yb,Ho,GdYTaO\(_4\) laser crystal [5] and Tm,Ho:LuYAG laser crystal [6] have been grown and proved that they can be used to generate the 2.911 \(\mu\text{m}\) laser. These examples show that the emission wavelength can be controlled by using different host materials and different doping ions together.

\(\text{Er}^{3+}\) is a very popular active ion since it has abundant energy levels. The near-infrared emission (~1.5 \(\mu\text{m}\)) and mid-infrared emission (~3.0 \(\mu\text{m}\)) are corresponding to the \(4\text{I}_{13/2} \rightarrow 4\text{I}_{15/2}\) and \(4\text{I}_{11/2} \rightarrow 4\text{I}_{13/2}\) transitions respectively. Also, \(\text{Er}^{3+}\) ion has a favorable absorption band with peaks around 970 nm. When the \(\text{Er}^{3+}\)-doping concentration of the host material is higher than 30%, the mid-infrared emission gets strong usually. In the same materials, when the concentration of doped-\(\text{Er}^{3+}\) is lower than the 20%, the emission wavelength tends to be the near-infrared, when comparing the high doped-\(\text{Er}^{3+}\) concentration. Because the lifetime of \(4\text{I}_{11/2}\) is an order of magnitude less than that of \(4\text{I}_{13/2}\), the particle inversion can hardly happen during the emission. This becomes the bottleneck of efficient laser operation in this wavelength. So, we seek to find a way to significantly shorten the lifetime of \(4\text{I}_{13/2}\). The high \(\text{Er}^{3+}\) doping concentration always shortens the lifetime of \(4\text{I}_{13/2}\), so it can promote the efficiency of the laser in this wavelength. That is to say, that high \(\text{Er}^{3+}\) doping is very advantageous for ~3.0 \(\mu\text{m}\) laser operation.
Furthermore, Er\textsuperscript{3+}-doped garnet crystals have tremendous potential in the mid-infrared field, because they have good thermal performance and high optical quality in this region. Among several Er\textsuperscript{3+}-doped garnet crystals, Er:YSGG shows a lower pumping threshold and higher conversion efficiency \cite{7–12}. In this work, our goal is to grow a series of 30% Er\textsuperscript{3+}:Y\textsubscript{3}(Sc\textsubscript{x}Ga\textsubscript{1−x})\textsubscript{5}O\textsubscript{12} crystals, and to explore how the proportion of Sc\textsuperscript{3+}/Ga\textsuperscript{3+} in the fibers can affect the fluorescence characteristics, and especially the emission wavelength at around \textasciitilde3.0 \textmu m. In the design of the experimental route, we controlled the different Y\textsubscript{3}Sc\textsubscript{5}O\textsubscript{12} (YSG) and Y\textsubscript{3}Ga\textsubscript{5}O\textsubscript{12} (YGG) proportion to change the host components with holding the Er\textsuperscript{3+} doped concentration as 30\%. Afterwards, we analyzed their spectral characteristics to check and compare the effect of the matrix.

In addition, the cost-effective \mu-PD method has been applied to explore the new crystals, as it has many advantages, such as the short growth cycle, saving raw materials, high component uniformity, and so on. So, it is a very promising technique for systematically studying the relationship between the fluorescence characteristics and proportion of Sc\textsuperscript{3+}/Ga\textsuperscript{3+} in the Y\textsubscript{3}(Sc\textsubscript{x}Ga\textsubscript{1−x})\textsubscript{5}O\textsubscript{12} crystals.

The ultimate goal of this study is to establish the relationship of the emission wavelength with the proportion of Sc\textsuperscript{3+}/Ga\textsuperscript{3+} in the Y\textsubscript{3}(Sc\textsubscript{x}Ga\textsubscript{1−x})\textsubscript{5}O\textsubscript{12} crystals, so that one can tune the lasing wavelength and improve the laser efficiency in 2.7–3.0 \textmu m easily in future.

2. Materials and Methods

The crystal fibers of 30\% Er\textsuperscript{3+}-doped compound of Y\textsubscript{3}(Sc\textsubscript{x}Ga\textsubscript{1−x})\textsubscript{5}O\textsubscript{12} have been grown by the \mu-PD technique with the Er\textsubscript{2}O\textsubscript{3}, Y\textsubscript{2}O\textsubscript{3}, Sc\textsubscript{2}O\textsubscript{3} and Ga\textsubscript{2}O\textsubscript{3} commercial powders as the starting materials. The Polycrystalline materials Y\textsubscript{2.1}Er\textsubscript{0.9}(Sc\textsubscript{x}Ga\textsubscript{1−x})\textsubscript{5}O\textsubscript{12} were made by Er\textsubscript{2}O\textsubscript{3}, Y\textsubscript{2}O\textsubscript{3}, Sc\textsubscript{2}O\textsubscript{3} and Ga\textsubscript{2}O\textsubscript{3} commercial powders that their purity is 99.99\% According to the different molar proportion of Sc\textsuperscript{3+}:Ga\textsuperscript{3+} (1.9, 2.8, 3.7 and 4.6) in every compound (see Table 1), the relevant powders were accurately weighed with keeping the total weight of every group in 10 g. Afterwards, these powders were adequately mixed for more than 48 h. Finally, we pressed them separately into tables. The solid-state reaction was applied to synthesize the polycrystalline materials according to the following formula:

\[
0.9\text{Er}_2\text{O}_3 + 2.1\text{Y}_2\text{O}_3 + 5\text{xSc}_2\text{O}_3 + 5\text{yGa}_2\text{O}_3 = 2\text{Y}_{2.1}\text{Er}_{0.9}(\text{Sc}_x\text{Ga}_y)_5\text{O}_{12} \quad (x + y = 1)
\]

| No. | x/y  | \text{Y}_{2.1}\text{Er}_{0.9}(\text{Sc}_x\text{Ga}_y)_5\text{O}_{12} |
|-----|------|--------------------------------------------------|
| 1   | 1:9  | \text{Y}_{2.1}\text{Er}_{0.9}(\text{Sc}_{0.1}\text{Ga}_{0.9})_5\text{O}_{12} |
| 2   | 2:8  | \text{Y}_{2.1}\text{Er}_{0.9}(\text{Sc}_{0.2}\text{Ga}_{0.8})_5\text{O}_{12} |
| 3   | 3:7  | \text{Y}_{2.1}\text{Er}_{0.9}(\text{Sc}_{0.3}\text{Ga}_{0.7})_5\text{O}_{12} |
| 4   | 4:6  | \text{Y}_{2.1}\text{Er}_{0.9}(\text{Sc}_{0.4}\text{Ga}_{0.6})_5\text{O}_{12} |

These tables were sintered at 1200 °C for 24 h to react completely and cooled down to room temperature within 18 h. The crystals were grown with the <111> direction YAG seeds and carried out in the home-made \mu-PD equipment with a pulling rates in the range of 1–2 mm/h, at a heating speed of RF power as 120 w/h and in a mixed atmosphere of argon and carbon oxide. Due to the ratios of Sc/Ga are different in every polycrystalline material, the growth processes were not the completely same. We found that the melting points of the polycrystalline materials changed, when the proportion of Sc/Ga altered. Correspondingly, we adjusted the heating power each time to fit the growth demand of the different component. Since every sample has different viscosity in the molten state, we controlled the pulling rates to keep the outflow speed of the melt so that we could maintain the high crystalline quality of every crystal. In addition, we adjusted the proportion of argon and carbon oxide so as to restrain the volatilization of Ga\textsubscript{2}O\textsubscript{3} and to protect the iridium crucible simultaneously. Finally, the four crystal fibers of different proportion of Sc/Ga, as shown in Figure 1, were grown for the
next measurements. In addition, as the proportion of YSG increases, the melting point of the crystal becomes higher and higher, and the difficulty of growth becomes larger and larger, and the obtained crystal is substantially completely cracked. Therefore, only the growth and test studies of these four ratios of samples are involved in this paper.

As-grown crystals were cut into disks with the faces perpendicular to <111> direction. And then, they were optically polished with 1mm thickness, as shown in Figure 2, for measuring their spectral properties.

![Figure 1](image1.jpg)  
**Figure 1.** The different ratios of YSG:YGG crystal fibers, 1:9 to 4:6 from left to right.

In order to verify the crystal structure and composition uniformity, the X-ray diffraction (XRD) patterns of the grown crystals were characterized by Bruker D8 ADVANCE (Bruker, Billerica, MA, USA). The concentrations of Y$^{3+}$, Sc$^{3+}$, Ga$^{3+}$ and Er$^{3+}$ were measured by X-ray Fluorescence spectrometry via Rigaku, ZSX-Primus II (Rigaku, Tokyo, Japan). The distribution and homogeneity of Er$^{3+}$ in the grown crystals were measured by electron-probe microanalyzer via SHIMADZU 1728 (Shimadzu, Kyoto, Japan). Next, the spectroscopy properties of the crystal samples were systematically studied. The absorption spectra of the samples were measured in the wavelength region of 900–1100 nm by the HITACHI U4100 UV-VIS-NIR Spectrophotometer (Hitachi, Tokyo, Japan). The mid-infrared (MIR) fluorescence spectra and decay curves were recorded in the wavelength region of 2450–3050 nm by Edinburgh Instruments FLS980 photoluminescence spectrometer (Edinburgh Instruments Ltd., Livingston, UK), by using 970 nm LD pumping source. The decay curves of the Er$^{3+}$:4$I_{13/2}$ level of the samples were recorded respectively at around 1530 nm emission band under excitation of 970 nm. The decay curves of Er$^{3+}$:4$I_{11/2}$ level of the samples were recorded respectively at around 2790 nm emission band under excitation of 970 nm. The RT Raman spectra were measured using a Horiba Jobin Yvon LabRAM HR Raman spectrometer (Horiba, Kyoto, Japan) equipped with a liquid N$_2$ cooled Ge detector.

3. Results

3.1. X-ray Fluorescence Spectrometry and EPMA Analyses

The concentrations of the Er$^{3+}$, Y$^{3+}$, Sc$^{3+}$ and Ga$^{3+}$ in the different samples have been measured. The data were shown in the Table 2. The concentration of doped-Er$^{3+}$ in every samples was around 30% actually, and the molar fraction ratios of Sc/Ga were almost equal to our predicted proportion.
Table 2. The concentration of the Er\(^{3+}\), Y\(^{3+}\), Sc\(^{3+}\) and Ga\(^{3+}\) in the different samples.

| YSG:YGG | Sc  | Ga  | Y   | Er  | Sc:Ga | Er%  |
|---------|-----|-----|-----|-----|-------|------|
| 1:9     | 0.076 | 0.670 | 0.305 | 0.133 | 1.8:82 | 30.4 |
| 2:8     | 0.118 | 0.545 | 0.352 | 0.152 | 2.8:24 | 30.2 |
| 3:7     | 0.225 | 0.502 | 0.364 | 0.135 | 3.6:69 | 29.1 |
| 4:6     | 0.309 | 0.416 | 0.369 | 0.145 | 4.5:93 | 29.2 |

Figure 3 showed the results of EPMA analyses, which indicated that the doped-Er\(^{3+}\) ions in every samples distribute uniformly, which was very important for the future application of crystal fibers in laser generation with high beam quality.

3.2. Spectroscopic Properties Analyses

The absorption spectra of all the samples have been measured at room temperature. We found that there was one absorption band among the absorption bands from 900 nm to 1100 nm in each spectrum corresponding to the ground state of \(^{4}I_{15/2}\) to the \(^{4}I_{11/2}\) multiples transitions of Er\(^{3+}\) ions. The results showed that the ratio of YSG:YGG compositions did not significantly change the wavelength of the absorption peak of the crystals. Here, the absorption spectra of the 1:9 sample was shown in Figure 4, as a representative.

Figure 4. The absorption spectra of the sample of YSG:YGG 1:9 at 900–1100 nm.
The mid-infrared fluorescence spectra of the samples were shown in Figure 5 in the range from 2450 nm to 3050 nm pumped by 970 nm LD source at room temperature. The spectra indicated that every measured sample had three emission peaks in the range of 2450–3050 nm, and they centered at about 2630 nm, 2710 nm and 2800 nm respectively due to 

$$\text{Er}^{3+}: {^4}\text{I}_{11/2} \rightarrow {^4}\text{I}_{13/2}$$

transitions. Comparing with the position of all emission peaks, we found an interesting regulation that when the proportion of Sc$^{3+}$ increased in the compounds, the emission peak of ~2800 nm would tend to be red-shifted. At the same time, another two emission peaks kept around the 2630 nm and 2710 nm. This phenomenon proved our assumption that we could adjust the emission wavelength with changing the proportion of Sc/Ga inside the $Y_{2.1}Er_{0.9}(Sc_xGa_{1-x})_3O_{12}$ crystals. According to our experiment results, the different proportion of Sc/Ga in the compound can affect the spectral properties of every crystal fiber. We analyzed the reason behind the phenomenon. It turned out as follows: the Sc$^{3+}$ and Ga$^{3+}$ are both in the octahedral site. The radius of Sc$^{3+}$ is bigger than the Ga$^{3+}$. With the proportion of Sc$^{3+}$ rising in the compound, the octahedral distortion also increases. The lattice parameter of the crystal fiber will change. So, the fluorescent properties and many other properties of the crystal will alter.

![Figure 5. The MIR fluorescence spectra of the samples of YSG:YGG from 1:9 to 4:6 at 2450–3050 nm.](image)

The Figure 6 simply demonstrates the procedure to obtain fluorescence lifetime. At first, the laser source send the pulsed light on the samples through the monochrometer. Secondly, the detector and oscilloscope turned the optical signals to electric signals on the screen. At last, this cycle was repeated many times so that we could obtain the decay curves.

![Figure 6. The Schematic of the test of fluorescence lifetime.](image)
The decay curves of the Er$^{3+}$: $^4I_{11/2}$ and $^4I_{13/2}$ levels in all the samples under excitation of 970 nm LD source were shown in Figures 7–9 respectively. The detected wavelengths were at 2790 nm and 1530 nm respectively. The fluorescence lifetime of these four crystal fibers were summarized in the Table 3. It can be found that the fluorescence lifetime of Er$^{3+}$: $^4I_{11/2}$ levels are one order of magnitude shorter than the Er$^{3+}$: $^4I_{13/2}$ levels. The Figure 7 show that the proportion of Sc/Ga can make little influence on the fluorescence lifetime of Er$^{3+}$: $^4I_{11/2}$ levels, and they all keep in the same magnitude. And in the Figures 8 and 9, the fluorescence lifetime of Er$^{3+}$: $^4I_{13/2}$ levels are shown. The fluorescent lifetime of Er$^{3+}$: $^4I_{13/2}$ levels can be affected by the proportion of Sc/Ga in the compounds largely. As compared, when the YSG increase in the crystal fiber, there is a trend that the fluorescence lifetime of Er$^{3+}$: $^4I_{13/2}$ level gets shorter. But interestingly the lifetime of Er$^{3+}$: $^4I_{13/2}$ levels is the shortest in those proportions as the proportion of YSG:YGG is 3:7. As we know, when the fluorescence lifetime of Er$^{3+}$: $^4I_{13/2}$ levels is significantly higher than the Er$^{3+}$: $^4I_{11/2}$ levels, it will cause the electron accumulation easily in the Er$^{3+}$: $^4I_{13/2}$ level, and that will lead to the lack of required electron inversion population. Finally, it will cause the ~3.0 µm electron transition self-termination [13]. This work proved that the proportion of YSG:YGG can considerably affect the fluorescence lifetime of Er$^{3+}$: $^4I_{13/2}$ levels, and it can be reduced most in the proportion of 3:7 in all current experimental data. This can help us to weaken the effect of electron transition self-termination by tuning the matrix composition, and get the laser of emission more efficiently in the ~3.0 µm. For the characteristic of the fluorescence lifetime, we found the proportion of Sc/Ga as the single variable in the experiment can explain the phenomenon. When the proportion of Sc$^{3+}$ raised in the compound, the crystal fiber showed the disordered state and the lattice distortion also increased. The different proportion of Sc/Ga changed the lattice parameter so that the decay curves of different crystal fibers turned out different results.

| Table 3. Fluorescence lifetime of different proportions of Sc:Ga from 1:9 to 4:6. |
|-------------------|---|---|---|---|
|                  | 1:9 | 2:8 | 3:7 | 4:6 |
| $^4I_{11/2}$     | 0.64 ms | 0.74 ms | 0.63 ms | 0.76 ms |
| $^4I_{13/2}$     | 6.04 ms | 4.32 ms | 1.95 ms | 3.72 ms |

Figure 7. Decay curves of Er$^{3+}$: $^4I_{11/2}$ levels in samples of YSG:YGG 1:9, 2:8, 3:7 and 4:6.
All samples showed a consistent pattern of YSGG. Therefore, the mixed crystals of YGG and YSG parameter changed a little and the octahedral distortion increased. Due to the change, the interplanar patterns. According to the formula 2

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3.3. XRD and Raman Analyses

Figure 10 shows the XRD patterns of every samples and YSGG standard for comparison. All samples showed a consistent pattern of YSGG. Therefore, the mixed crystals of YGG and YSG did not cause a change in the crystal phase, and they all maintained the structural characteristics of the garnet series crystals. We can see that all the peaks transit slightly to the left in the XRD patterns.

Figure 8. Decay curves of \( \text{Er}^{3+}:I_{13/2} \) levels in samples of YSG:YGG 1:9, 2:8 and 4:6.

Figure 9. Decay curves of \( \text{Er}^{3+}:I_{11/2} \) levels in samples of YSG:YGG 3:7.

\[ Crystals \ 2019, 9, x \]

According to the formula 2

\[ 2\sin\theta = n\lambda \]

the interplanar spacing “d” becomes bigger with the “θ” decreasing. The radius of Sc\(^{3+}\) is bigger than the Ga\(^{3+}\). With proportion of Sc\(^{3+}\) rising, the lattice parameter changed a little and the octahedral distortion increased. Due to the change, the interplanar spacing increased, too. So, the peaks of the XRD Patterns transit slightly to the left.
The Raman spectra of those samples and the maximum Raman shift of every sample have been shown in the Figure 11. The spectra indicated that the different proportion of YSG:YGG made little influence on non-radiative transitions. The maximum Raman shift of every sample was almost the same, and all the peaks were nearly centered at about 895 nm.

4. Conclusions

In order to explore the influence of components on the Er\textsuperscript{3+} emission wavelengths, the series crystal fibers of 30\% Er\textsuperscript{3+}-doped YSG:YGG have been grown by using the \(\mu\)-PD technique. As the concentration of doped-Er\textsuperscript{3+} keeping constant, the proportion of YSG:YGG in the compounds clearly affected the emission properties of crystal fiber. With changing the proportion of YSG:YGG, the emission peak wavelengths could be tuned in ~3.0 \(\mu\)m region. The fluorescence lifetime of Er\textsuperscript{3+}: 

**Figure 10.** The XRD patterns of the samples of YSG:YGG 1:9, 2:8, 3:7, 4:6 and YSGG standard for comparison.

**Figure 11.** Raman spectra of Er\textsuperscript{3+}-doped YSG:YGG from 1:9 to 4:6.
4I_{13/2} levels decreased greatly by increasing the proportion of YSG properly in the crystal fibers, in the meantime the fluorescence lifetime of Er^{3+}\rightarrow I_{11/2} levels basically remained the same. This proved that the self-termination of the 4I_{11/2}\rightarrow 4I_{13/2} electron transition could be solved by changing the YSG:YGG in the host materials. More importantly the finer and larger range of constituent adjustments and their effects on crystal emission spectra are for further study.

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