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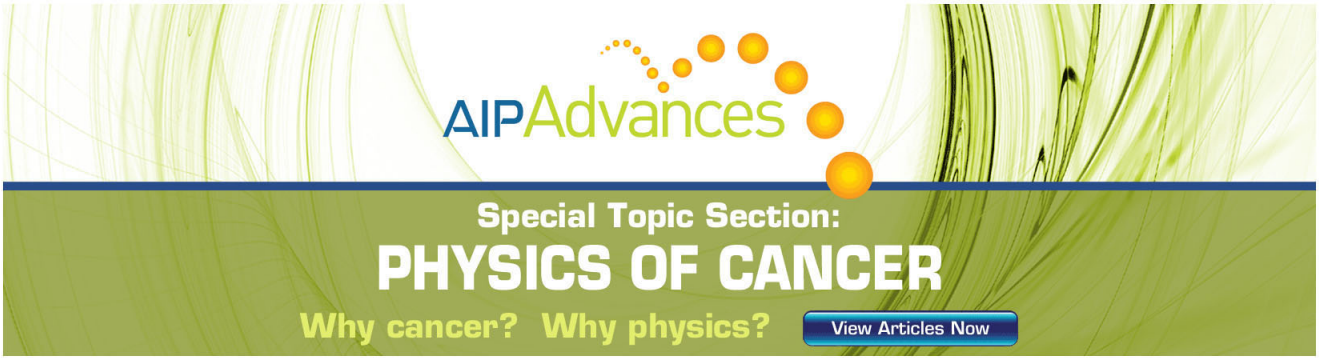


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# Demonstration of enhanced population feeding of the 1.53 $\mu$m emitting level of $Er^{3+}$ in $TeO_2-WO_3-Li_2O-P_2O_5$ glasses using upconversion luminescence spectroscopy

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$P_2O_5$ is introduced into the $Er^{3+}$-doped tungsten tellurite glasses to speed up the $^4I_{11/2}$-$^4I_{13/2}$ relaxation. The properties of infrared to visible upconverted luminescence as a function of $P_2O_5$ contents are studied to demonstrate the enhanced feeding rate of the 1.53 $\mu$m emitting level of $Er^{3+}$, which is in good agreement with that obtained using infrared emission spectra. The contributions of excited state absorption and cooperation energy transfer to the red and green upconverted luminescences are quantitatively evaluated. The $Er^{3+}$-doped telluride glasses containing $P_2O_5$ could be a promising candidate as a host for broadband erbium-doped fiber amplifiers. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2891788]

## I. INTRODUCTION

$Er^{3+}$-doped telluride glasses have been extensively investigated for its potential use in broadband erbium-doped fiber amplifiers (EDFAs) in the 1.53 $\mu$m window of optical communication.[1–5] They have exhibited a wide 1.53 $\mu$m emission band, a large stimulated emission section, and a high emission efficiency.[3] However, as a host for EDFA, telluride glass has a drawback that the phonon energy of the glass is as low as 770 $cm^{-1}$, which leads to a slow population feeding of the 1.53 $\mu$m emitting level, $^4I_{13/2}$, through the nonradiative $^4I_{11/2}$-$^4I_{13/2}$ relaxation upon pumping into the $^4I_{11/2}$ level of $Er^{3+}$ at 980 nm. In this case, an intense green upconverted luminescence (UCL) originating from $^2H_{11/2}+{}^4S_{3/2}\rightarrow{}^4I_{15/2}$ transitions and a red one from the $^4F_{9/2}\rightarrow{}^4I_{15/2}$ transition of $Er^{3+}$ are generated by depopulating the $^4I_{11/2}$ and $^4I_{13/2}$ levels, strongly suppressing the optical pumping efficiency for the 1.53 $\mu$m emission. To overcome this drawback, Shen *et al.*[6] added $WO_3$ with phonon energy of 920 $cm^{-1}$ to form tungsten tellurite (TW) glasses, in which the nonradiative relaxation rate was increased. Cho *et al.* selected $B_2O_3$ as an additive,[7] remarkably speeding up the nonradiative relaxation. Unfortunately, the emission efficiency of the $^4I_{13/2}$ level also decreased quickly with increasing $B_2O_3$ contents because the B–O bond has a very large vibronic energy of about 1500 $cm^{-1}$. Therefore, an additive with appropriate phonon energy is required. In our previous work,[8] we have selected $P_2O_5$ as an additive introduced into $Er^{3+}$-doped TW glasses. The study of near infrared emission spectra demonstrates that the nonradiative relaxation was further speeded up by a factor of 3.3 without reduction of the quantum efficiency for the 1.53 $\mu$m emission because the P–O bond has a satisfied vibronic energy of about 1200 $cm^{-1}$.

In this paper, we demonstrate the fast population feeding of the 1.53 $\mu$m emitting level of $Er^{3+}$ in $TeO_2-WO_3-Li_2O-P_2O_5$ (TWP) glasses using UCL spectroscopy. The reduction of the UCL with increasing $P_2O_5$ contents indicates that the $^4I_{11/2}$-$^4I_{13/2}$ nonradiative relaxation rate is enhanced and its value is determined, which are in good agreement with those obtained using nearinfrared emission spectra. The dynamical processes in UCL under 980 nm and/or 808 nm excitation are systematically studied for different $P_2O_5$ content. The contributions of excited state absorption (ESA) and cooperation energy transfer (ET) to the red and green UCLs are quantitatively evaluated.

## II. EXPERIMENTAL

$Er^{3+}$-doped glasses, $(40-0.4x)TeO_2-(30-0.3x)WO_3-(30-0.3x)Li_2O-xP_2O_5$ $(x=0,2,4,6)$ $(TWP_x)$, with $Er^{3+}$ concentration of 1.0 mol % and a glass $TWP_0$:0.1 mol % $Er^{3+}$ were prepared using the conventional melting and quenching method. Photoluminescence (PL) and UCL spectra were measured using a Hitachi F4500 spectrometer. An 808 nm emitting laser diode (LD) and a 980 nm emitting LD are used as pumping sources for UCL. Absorption spectra were collected using an UV-3101PC spectrometer. The lifetime of the $^4I_{13/2}$ level was recorded with a 500 MHz Tektronix digital oscilloscope under excitation of 532 nm pulses from an yttrium aluminum garnet laser.

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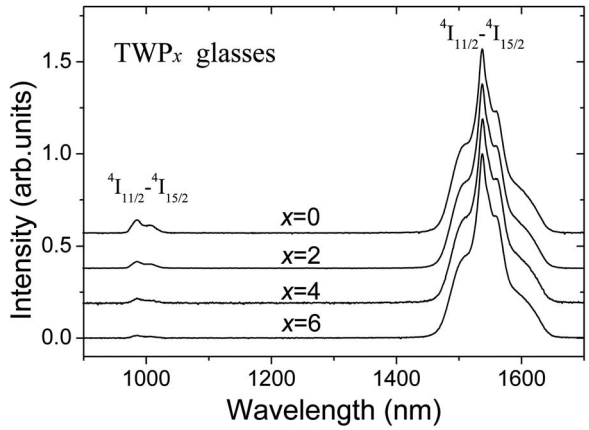


FIG. 1. The infrared emission spectra of $Er^{3+}$ in TWP samples under 808 nm excitation; spectra are normalized to the intensity of 1.5 $\mu$m emission.

## III. RESULTS AND DISCUSSION

### A. Determination of the nonradiative $^4I_{11/2}$-$^4I_{13/2}$ relaxation rates by using near infrared emission spectra in $Er^{3+}$-doped TWP glasses with various $P_2O_5$ contents

Figure 1 shows the near infrared emission spectra of $Er^{3+}$ in TWP glasses under 808 nm excitation. The width of the 1.53 $\mu$m band is about 65 nm, which has been affected little with $P_2O_5$ addition. It can be seen that the integrated intensity ratio ($\alpha$) of the 1.53 $\mu$m band to the 0.98 $\mu$m band increases as $P_2O_5$ content increases. As described in our previous work,[8] this ratio has a relationship with the $^4I_{11/2}$-$^4I_{13/2}$ nonradiative relaxation rate $W_{21}$, i.e., $W_{21}=\alpha A_{21}/A_{10}\tau_1$, where $A_{21}$ and $A_{10}$ are the radiative rates of $^4I_{11/2}$-$^4I_{13/2}$ and $^4I_{13/2}$-$^4I_{15/2}$ transitions, respectively; $\tau_1$ is the lifetime of the $^4I_{13/2}$ level. We have calculated radiative transition rates by analyzing their absorption spectra (Fig. 2) using the Judd–Ofelt (JO) theory[9,10] and found that these optical parameters are hardly influenced by the addition of $P_2O_5$. Thus, the increase of $\alpha$ after $P_2O_5$ addition is the result of the enhanced $^4I_{11/2}$-$^4I_{13/2}$ nonradiative relaxation rate due to phonon energy modification by $P_2O_5$.

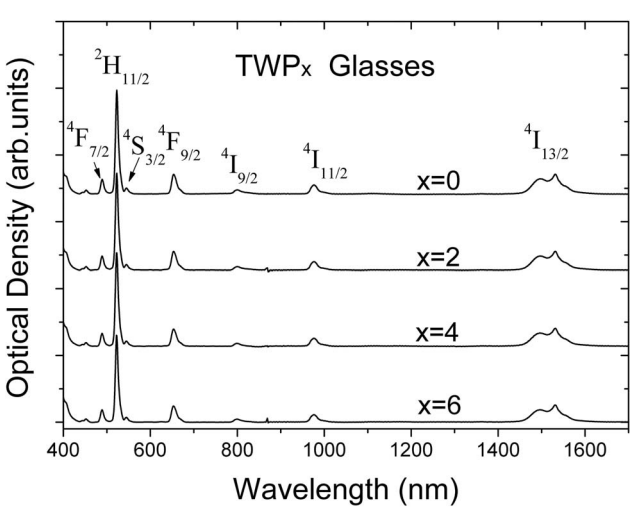


FIG. 2. Absorption spectra of TWP glasses doped with 1.0 mol % of $Er^{3+}$.

TABLE I. Parameters ($W_{21}$, $\tau_1$, $\tau_2$, $\tau_5$) determined from different methods, normalized to the one of $TWP_0$ sample.

| Parameters | | Samples | | | |
|---|---|---|---|---|---|
| | | $TWP_0$ | $TWP_2$ | $TWP_4$ | $TWP_6$ |
| $W_{21}$ | $\alpha$ | 1 | 1.67 | 2.63 | 3.37 |
| | $I_{G808}/I_{G980}$ | 1 | 1.53 | 2.56 | 3.33 |
| | $I_{G980}/I_{G380}$ | 1 | 1.5 | 2.5 | 3.24 |
| $\tau_1$ | Time decay | 1 | 1 | 1.03 | 1.06 |
| | $I_{G808}/I_{G380}$ | 1 | 1.02 | 1.02 | 1.03 |
| $\tau_5$ | $I_{G380}$ | 1 | 0.66 | 0.40 | 0.33 |
| $\tau_2$ | $1/W_{21}(\alpha)$ | 1 | 0.60 | 0.38 | 0.30 |

The $W_{21}$ rates calculated by $\alpha$ and the $\tau_1$ determined by luminescence time decay measurement are listed in Table I. The value of $W_{21}$ in $TWP_6$ glass is about 3.3 times higher than that in $TWP_0$ glass. It should be noted that the lifetime of the $^4I_{11/2}$ level ($\tau_2$) is determined by $1/W_{21}$ because the radiative transition rate of the $^4I_{11/2}$ level is much smaller than $W_{21}$. All the data listed in Table I are normalized to the corresponding values from $TWP_0$ glass.

### B. Determination of the nonradiative $^4I_{11/2}$-$^4I_{13/2}$ relaxation rates using UCL in $Er^{3+}$-doped TWP glasses with various $P_2O_5$ contents

Figures 3(a) and 3(b) illustrate the UCL spectra of $Er^{3+}$ in TWP glasses with different $P_2O_5$ contents under the 980 and 808 nm LD excitations, respectively. Three emission bands centered at 524, 546, and 655 nm are observed and assigned to the transitions to $^4I_{15/2}$ from $^2H_{11/2}$, $^4S_{3/2}$, and $^4F_{9/2}$, respectively. Clearly, the red UCL at 655 nm is relatively weaker than the green, and all the UCL intensities decrease with increasing $P_2O_5$ contents. Importantly, the UCL under 980 nm excitation decreases faster than that under 808 nm excitation. One can observe that the intensity ratios of the red to the green emissions ($I_R/I_G$) do not change

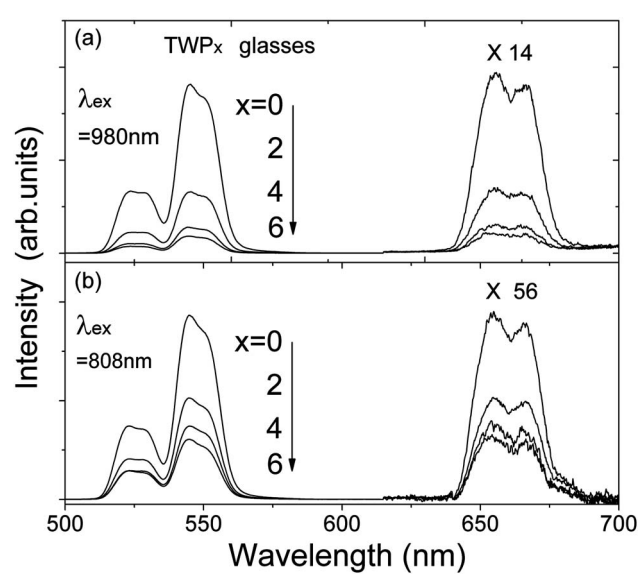


FIG. 3. UCL spectra of $Er^{3+}$ in TWP glasses under the excitations of 980 and 808 nm.

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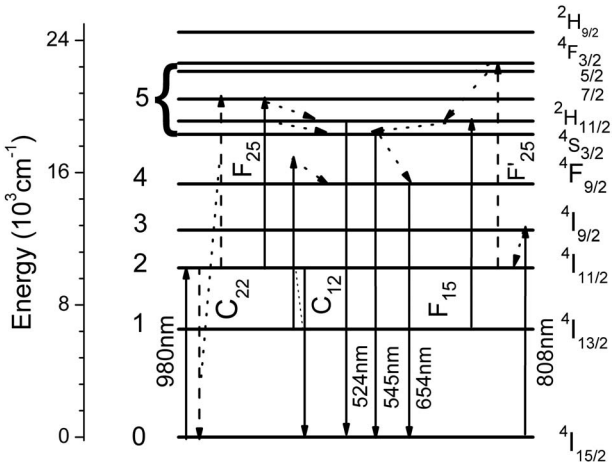


FIG. 4. Energy level diagram of $Er^{3+}$ ions under excitations of 980 nm/808 nm.

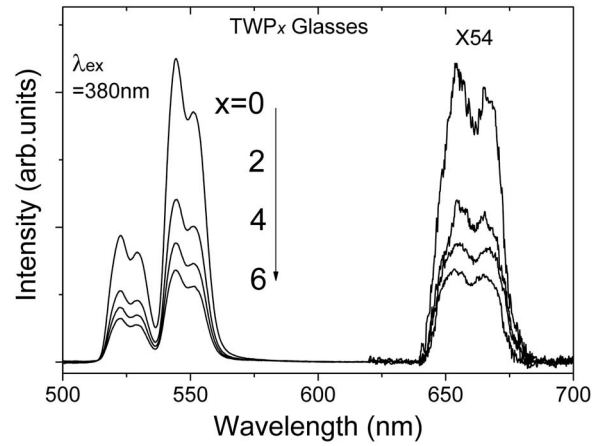


FIG. 5. Emission spectra of $Er^{3+}$ in TWP glasses under the excitation of 380 nm.

with increasing $P_2O_5$ contents. Moreover, the value of the ratio upon 980 nm excitation is about four times as high as that upon 808 nm excitation.

The upconversion processes of $Er^{3+}$ include the ESA and cooperation ET.[11–17] The possible upconversion mechanisms upon excitation of 980 nm and/or 808 nm are illustrated in the energy level diagram of $Er^{3+}$, as shown in Fig. 4. Under steady excitation by 980 nm, we have the following rate equations:

$$dn_1/dt = (A_{21} + W_{21})n_2 - n_1/\tau_1 = 0, \quad (1)$$

$$dn_2/dt = F_{02}n_0 - n_2/\tau_2 = 0, \quad (2)$$

$$dn_4/dt = W_{54}n_5 + C_{12}n_1n_2 - n_4/\tau_4 = 0, \quad (3)$$

$$dn_5/dt = C_{22}n_2^2 + F_{25}n_2 - n_5/\tau_5 = 0, \quad (4)$$

and for 808 nm excitation, we have

$$dn_1/dt = (A_{21} + W_{21})n_2 - n_1/\tau_1 = 0, \quad (5)$$

$$dn_2/dt = (W_{32} + A_{31})n_3 - n_2/\tau_2 = 0, \quad (6)$$

$$dn_3/dt = F_{03}n_0 - n_3/\tau_3 = 0, \quad (7)$$

$$dn_4/dt = W_{54}n_5 + C_{12}n_1n_2 - n_4/\tau_4 = 0, \quad (8)$$

$$dn_5/dt = C_{22} + n_2^2 + F'_{25}n_2 + F_{15}n_1 - n_5/\tau_5 = 0, \quad (9)$$

where $n_i$ and $\tau_i$ are the population and lifetime of the $i$th level, respectively. $W_{ij}$ is the nonradiative transition rate from the $i$th level to the $j$th level, $F_{ij}$ is the pumping rate from the $i$th level to the $j$th level, $C_{ij}$ is the coefficient for ET between an ion in the $i$th level and another one in the $j$th level, and $A_{ij}$ is the radiative transition rate of the $i$th level to the $j$th level. In the present glass, $W_{ij}$ (except $W_{10}$) is much larger than the total radiative transition rate of the $i$th level; the lifetime of the $i$th energy level is determined by $1/W_{ij}$.

Using Eqs. (1)–(9), the green UCL intensities under 980 and 808 nm excitations can be, respectively, written as

$$I_{G980} = A_{50}n_5 = A_{50}\tau_5(F_{25}F_{02}n_0\tau_2 + C_{22}F_{02}^2n_0^2\tau_2^2) \quad (10)$$

and

$$I_{G808} = A_{50}n_5 = A_{50}\tau_5(F_{15}F_{03}n_0\tau_1 + F'_{25}F_{03}n_0\tau_2 + C_{22}F_{03}^2n_0^2\tau_2^2). \quad (11)$$

If we consider the radiative transition rates to be independent of $P_2O_5$ contents based on the analysis by the JO theory and keep the excitation intensity constant, only the variations of $\tau_i$ $(i=1,2,5)$ with $P_2O_5$ contents are of interest. The values of $\tau_5$ are obtained by measuring the intensities of the green emission upon 380 nm excitation into level 5. Under steady excitation at 380 nm, one has $n_5/\tau_5 = F_{05}n_0$, so the intensity of the green band ($I_{G380}$) is the measure of $\tau_5$. Figure 5 shows the PL spectra of $Er^{3+}$ in TWP glass with different $P_2O_5$ contents under 380 nm excitation, exhibiting the reduction of the green band with increasing $P_2O_5$ contents. The values of $\tau_5$ are determined from the PL spectra in Fig. 5 and listed in Table I. If we denote the $\tau_1$ that was determined from time decay measurements by $\tau_1$(TD) and the $\tau_2$ from intensity ratio measurements by $\tau_2(\alpha)$, ideal linear relationships are observed between $I_{G980}$ and $\tau_2(\alpha)\tau_5$, and between $I_{G808}$ and $\tau_1(\mathrm{TD})\tau_5$, as shown in Figs. 6(a) and 6(b), respectively. As the result, only the first term on the right

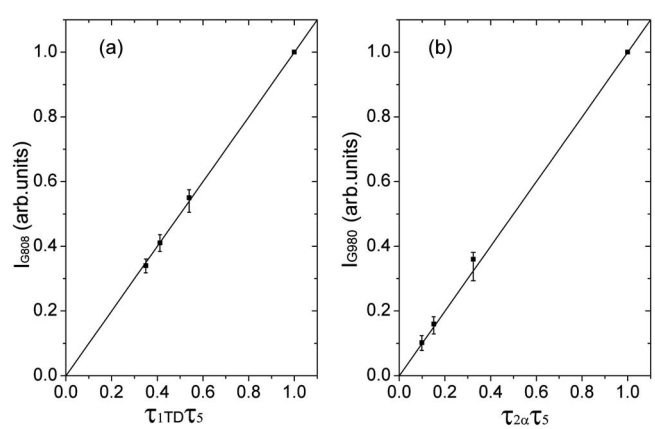


FIG. 6. A plot of $I_{G980}$ vs $\tau_2(\alpha)\tau_5$, and $I_{G808}$ vs $\tau_1(\mathrm{TD})\tau_5$, analyzed using linear curve fit.

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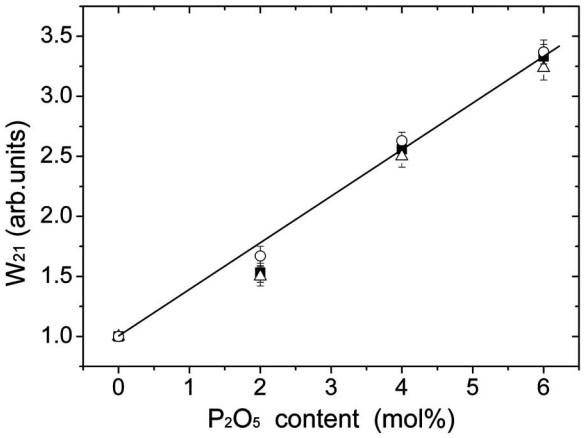


FIG. 7. A comparison of $W_{21}$ determined by ratios $\alpha$, $\tau_5/I_{G980}$, and $I_{G808}/I_{G980}$ as a function of $P_2O_5$ contents.

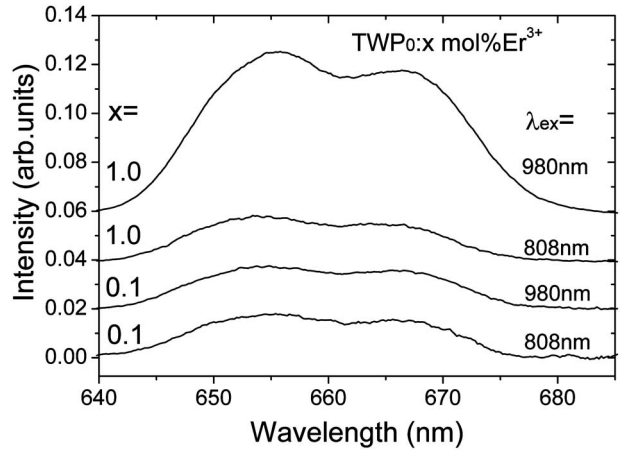


FIG. 8. Red UCL spectra of $TWP_0$ samples with 0.1 and 1 mol % $Er^{3+}$ under the excitations of 980 and 808 nm; each one is normalized to the intensity of green emission.

side of Eqs. (10) and (11) play the dominant role on the green UCL. This reveals that the ET process involving interaction between two ions has a negligible contribution to the green UCL for both 980 and 808 nm excitations at the present doping concentration of $Er^{3+}$ in this work, and the green UCL is dominantly governed by the ESA process of the $^4I_{11/2}$ level ($F_{25}$) for 980 nm excitation and the $^4I_{13/2}$ level ($F_{15}$) for 808 nm excitation.

In this case, from Eqs. (10) and (11), we rewrite

$$I_{G980} = A_{50}F_{25}F_{02}n_0\tau_2\tau_5, \tag{10'}$$

$$I_{G808} = A_{50}F_{15}F_{03}n_0\tau_1\tau_5. \tag{11'}$$

From Eqs. (10′) and (11′), the $W_{21}(=1/\tau_2)$ can be obtained by $\tau_5/I_{G980}$ or also by $I_{G808}/I_{G980}$ due to almost unchanged $\tau_1$. Figure 7 gives a comparison of $W_{21}$ determined by ratios $\alpha$, $\tau_5/I_{G980}$, and $I_{G808}/I_{G980}$ as a function of $P_2O_5$ contents. Obviously, these $W_{21}$ determined by different measurements are in good agreement with each other and have a linear relationship with $P_2O_5$ contents. The value of $W_{21}$ in $TWP_6$ glass is about 3.3 times higher than that in $TWP_0$ glass. $\tau_1$ can be also obtained by $I_{G980}/\tau_5$ using Eq. (11′). These $W_{21}$ and $\tau_1$ determined by different measurements are listed in Table I. Now we focus on the relative intensities of the red UCL to the green ($I_R/I_G$) upon 980 and 808 nm excitations. Using Eqs. (1)–(9), (10′), and (11′), we have the simplified expressions of the ratio $I_R/I_G$ for the different wavelength excitations,

$$\frac{I_R}{I_G}(980) = \frac{\tau_4 A_{40}}{\tau_5 A_{50}}\left(W_{54}\tau_5 + \frac{C_{12}F_{02}\tau_1 n_0}{F_{25}}\right), \tag{12}$$

$$\frac{I_R}{I_G}(808) = \frac{\tau_4 A_{40}}{\tau_5 A_{50}}\left(W_{54}\tau_5 + \frac{C_{12}F_{03}\tau_2 n_0}{F_{15}}\right). \tag{13}$$

The first terms on the right side of Eqs. (12) and (13) are the contributions by the multiphonon relaxation from the green emitting levels, $^4S_{3/2}$ (level 5), to the red emitting level, $^4F_{9/2}$ (level 4), and the second terms are the cooperation ETs, which are $Er^{3+}$ concentration dependent. Figure 8 shows the comparison of the red UCL spectra in $TWP_0$ glasses doped with 1 and 0.1 mol % $Er^{3+}$ upon 980 and 808 nm excitations, where the spectral intensity is normalized to the green UCL. It is observed that the relative intensities of the red UCL for 980 nm excitation is stronger than that for 808 nm excitation in $TWP_0$ glass doped with 1 mol % $Er^{3+}$. This phenomenon can be explained well using Eqs. (12) and (13). It is known that $\tau_1$ is two orders of magnitude larger than $\tau_2$,[4] and $F_{ij}$ are in the same order of magnitude; the second term on the right side of Eq. (12) is thereby larger than that of Eq. (13), i.e., $C_{12}F_{02}\tau_1 n_0/F_{25} \gg C_{12}F_{03}\tau_2 n_0/F_{15}$. When decreasing $Er^{3+}$ concentration ($n_0$) from 1 to 0.1 mol %, the relative intensities of the red UCL for 808 nm excitation is unchanged, as shown in Fig. 8, implying that the first term on the right side of Eq. (13) is larger than the second term for $Er^{3+}$ concentration by at least less than 1 mol %. Moreover in Fig. 8, the red UCL for 980 nm excitation is about four times stronger than that for 808 nm excitation for $Er^{3+}$ concentration of 1 mol % and decreases close to that for 808 nm excitation as $Er^{3+}$ concentration is reduced to 0.1 mol %. This indicates that the second term on the right side of Eq. (12) is indeed concentration dependent and about three times larger than the first term for $Er^{3+}$ concentration of 1 mol %.

## IV. CONCLUSIONS

The introduction of $P_2O_5$ into the $Er^{3+}$-doped tungsten tellurite glasses speeds up population feeding of the 1.53 $\mu$m emitting level, $^4I_{13/2}$, through nonradiative $^4I_{11/2}$-$^4I_{13/2}$ relaxation and simultaneously suppresses UCL without affecting the emission efficiency of the $^4I_{13/2}$ level. The enhanced rate by a factor of 3.3 for $P_2O_5$ content of 6% is obtained using UCL spectroscopy, which is in good agreement with that determined using infrared emission spectra. The green UCL is dominantly governed by the ESA process at the present doping concentration of $Er^{3+}$. The contribution of cooperative ET process to the red UCL is much bigger for 980 nm excitation than that for 808 nm excitation. In UCL of TWP glasses with $Er^{3+}$ concentration of 1 mol %, the red emitting level is dominantly populated by multiphonon relaxation from the green emitting levels for 808 nm excitation and by cooperative ET as well as a small contribution from the mul-

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tiphonon relaxation for 980 nm excitation. The red/green UCL ratio in $Er^{3+}$-doped TWP glasses is controllable by adjusting excitation wavelength and doping concentrations. The telluride glass with $P_2O_5$ addition is a promising candidate as a host for broadband EDFAs with good performance pumping at 980 nm.

## ACKNOWLEDGMENTS

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