1. Introduction

Optical temperature sensing technologies have received much attention in the light of their potential applications in daily life, scientific research, industrial production and other fields, owing to its excellent characteristics such as fast response, high sensitivity, and high resolution [1–3]. In the environment of non-contact, radiation resistance, strong magnetic field and other extreme conditions, high accuracy temperature measurement technology is very important. The technology based on the fluorescence intensity ratio of rare earth ions is more suitable for temperature measurements in extreme environments [4] than the fluorescence intensity or fluorescence lifetime methods, because it is not affected by pumping disturbance and fluorescence loss [5, 6]. The temperature measurement technology of fluorescence intensity ratio is mainly based on two thermally-coupled energy levels (200 ~ 2000 cm⁻¹) that comply with the Boltzmann distribution of rare earth ions [3, 6, 7]. The fluorescence intensity ratio emitted by energy levels changes with temperature to achieve temperature measurement [8]. Therefore, the research of temperature sensing performance based on FIR technology has a great application prospect.

In recent years, the field of optical temperature sensing has focused on rare earth doped upconversion luminescent materials. Huang et al. found that the sensitivity of Er³⁺/Yb³⁺ co-doped TeO₂–AlF₃–NaF–BaF₂–ErF₃–YbF₃ glass reached a maximum of 54.09 x 10⁻⁴ K⁻¹ at 531 K [2]. Prasenjit et al. reported the maximum sensitivity of Er³⁺/Yb³⁺ co-doped Sb₂O₃–WO₃–Li₂O glass-ceramic sample at 320 K was 0.0119 K⁻¹ [9]. Yuyuan et al. found that Er³⁺/Yb³⁺ co-doped TeO₂–WO₃–La₂O₃–Na₂O glasses had a very wide measurement temperature range of 285 to 563 K and a maximum sensitivity of 86.7 x 10⁻⁴ K⁻¹ at 553 K [10]. The research on temperature sensing based on upconversion luminescent materials is flourishing. Developing new materials and improving sensitivity is still the research goal. For some non-radiative transition energy transfers assisted by multi-phonons, a higher matrix phonon energy results in a
higher non-radiative transition rate \[10, 11\], reducing the upconversion luminescence efficiency. Therefore, it is important to choose a suitable glass matrix material with low phonon energy. New heavy metal oxide glasses have low phonon energy compared to other materials, improving the radiative transition efficiency of rare earth ions. It is worth noting that TiO\(_2\)-based glasses has excellent mechanical strength, thermal stability, corrosion resistance, and good solubility of rare earth ions suitable as a matrix material \[12\].

Researchers have strongly considered rare earth ions because of the unique characteristic that 4f electrons are shielded by outer 5S\(_2\) and 5P\(_6\) electron shells and are less affected by the external environment \[13\]. The luminescence of rare earth ions mainly comes from the electron transition between the 4f levels, Rare earth ions have abundant electron transition levels \[14\] and long lifetime excited states \[15\], which are ideal for upconversion luminescent materials as activators and sensitizers. The luminescent ion Er\(^{3+}\) has high luminous efficiency, while the sensitized ion Yb\(^{3+}\) has a large absorption cross section at 980 nm \[2, 14–18\]. Therefore, Er\(^{3+}/\)Yb\(^{3+}\) co-doped glasses can improve the absorption of the material for the pump laser. The chemical properties and electronic structures of La\(^{3+}\) and rare earth luminescent ions are similar, allowing doping of the luminescent ions in the matrix material to high concentrations. The maximum phonon energy of ZrO\(_2\) is only 470 cm\(^{-1}\) \[19\], and its addition to the matrix reduces the phonon energy of the matrix material. Therefore, TiO\(_2\)–ZrO\(_2\)–La\(_2\)O\(_3\) was selected as the matrix material in this research.

Commonly, the difference \(\Delta T\) between the starting crystallization temperature of glass \(T_o\) and the glass transition temperature \(T_g\) can be used to characterize glass forming ability \[20, 21\]. Nevertheless, the formation ability of TiO\(_2\)-based glasses is relatively low (\(~\)80 °C), TiO\(_2\)-based melts tend to crystallize during solidification and require a very high cooling rate to form glass. So it is difficult to prepare bulk glasses by traditional high temperature melting methods \[22\]. Containerless technologies such as aerodynamic levitation technology can restrain the heterogeneous nucleation \[23\], allowing for deep undercooling and rapid solidification \[24, 25\] and can be used to prepare glasses with low formation ability.

In this work, Er\(^{3+}/\)Yb\(^{3+}\) co-doped TiO\(_2\)–ZrO\(_2\)–La\(_2\)O\(_3\) glasses were prepared by an aerodynamic levitation method. The density of the glass was determined, and the transmittance before and after irradiation was studied. The absorption and emission cross sections at 525 nm were calculated. Temperature sensitivity was studied systematically. The crystal-phase structure of glass samples was ascertained by x-ray diffractometer (XRD). The morphology of the micro-crystal was obtained by Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) and Selective Electron Diffraction (SAED). Finally, resistance to etching under acid and alkali environments was researched to determine the suitability of the glass under extreme conditions.

2. Experimental

2.1. Aerodynamic levitation device

Aerodynamic levitation device generally includes laser heating system, air suspension system, cooling water system, real-time observation system, parameter control system. The schematic structure is shown in figure 1.

![Figure 1. The diagram of the aerodynamic levitation system.](image-url)
control the gas flow and laser power and is the key equipment to adjust the sample suspension state and preparation.

2.2. Preparation and characterization
The composition of the glass is \((\text{La}_{0.78}\text{Er}_{0.06}\text{Yb}_{0.16})(\text{Ti}_{0.95}\text{Zr}_{0.05})_{2.25}\text{O}_6\) \((99.99\%), \text{TiO}_2 \((99.99\%), \text{ZrO}_2 \((99.9\%), \text{Er}_2\text{O}_3 \((99.99\%) \text{and Yb}_2\text{O}_3 \((99.99\%)) \text{powders were mixed completely with alcohol. The mixture was}

pressed into a tablet, and then sintered in a muffle furnace at 1200 °C for 10 h. The samples suspended in an \(\text{O}_2 \) stream on a nozzle and melted in a laser heating furnace. The samples were held at a steady state stream and temperature for 1 min to allow for homogenization, and then the laser was turned off. Samples with a diameter of 3.4 mm were successfully prepared, which were then reduced to a thickness of 1.5 mm by double-sided polishing for follow-up test.

The density of glass was measured by an automatic true density analyzer (3H-2000TD1). The transmittance of glass before and after \(\gamma\)-ray irradiation was obtained by using Varian Cary 5000 M. The upconversion fluorescence spectra were recorded by Fluorescence Spectrophotometers (F-4600, Hitachi, Japan) in the temperature range of 298 K to 498 K. The glass-ceramics were prepared at the heating temperatures of 875, 900 and 925 °C for 50 min. The crystal-phase structure of glass samples was ascertained by an x-ray diffractometer (D8 DISCOVER). The morphology features and the nanocrystal size of samples were measured by SEM (Verios G4) and TEM (Tecnai G2 F20).

The acid resistance was tested by placing the glass sample into a 150 ml HCl solution (mass fraction = 36%–38%) and etching it at 200 °C for 50 h. The sample was washed with deionized water, cleaned with alcohol in an ultrasonic sink for 30 min, and dried. For alkaline conditions, the procedure was repeated using an NaOH solution with a mass fraction of 33%. The relative mass loss was calculated, and the etched surface morphology was examined by SEM to determine the acid and alkali resistance of the glass.

3. Results and discussion

3.1. Refractive index
The density of the \((\text{La}_{0.78}\text{Er}_{0.06}\text{Yb}_{0.16})(\text{Ti}_{0.95}\text{Zr}_{0.05})_{2.25}\text{O}_6\) glass was 5.346 ± 0.224 g cm\(^{-3}\) as measured by the automatic true density analyzer. The density of this glass is higher than that of \(\text{La}_2\text{O}_3–\text{TiO}_2–\text{Nb}_2\text{O}_5\) glasses, which is 5.255 g cm\(^{-3}\) [26], indicating that the glass has a higher refractive index.

Figure 2 shows the relationship between refractive index and incident wavelength. The refractive index at 587.56 nm is used to characterize the refractive properties of the material. The refractive index of the glass reached 2.30. A high refractive index improves the radiative transition probability of luminescent ions [27], which results in a stronger upconversion luminescence. Wavelength dispersion is an important optical property of glass. Abbe number \((V_d)\) can be used to characterize the dispersion ability of optical glass. The larger the Abbe number, the smaller the dispersion. Abbe number \((V_d)\) can be calculated as [28]:

\[
V_d = \left(\frac{n_d - 1}{n_f - n_c}\right)
\]
Where $n_d$, $n_f$, and $n_c$ are the refractive indices corresponding to 587.56, 486.10 and 656.30 nm respectively [27], corresponding values are 2.30, 2.35 and 2.28 respectively. The $V_d$ of the glass calculated is 18.57. Generally, $V_d$ of commercial optical glasses is in the ranges of 15–100 [28], So the glass performs excellent optical properties.

### 3.2. Transmittance spectra

The transmittance spectra of glasses in the range of 300 to 2100 nm are shown in figure 3. Except the characteristic absorption of Er$^{3+}$ and Yb$^{3+}$, the transmittance reached 70% in the visible region. At 455, 490, 525, 545 and 655 nm, five characteristic absorption bands correspond to the transitions from ground state of Er$^{3+}$ to excited state energy levels of $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{15/2}$, $^4S_{3/2}$ and $^4F_{9/2}$, respectively. In the near infrared band, the transmittance of glass reached a maximum of 75.46%. There are three characteristic absorption peak centers at 795, 980 and 1530 nm, which are the transitions from the ground state of Er$^{3+}$ to $^4I_{9/2}$, $^2F_{5/2}$ of Yb$^{3+}$ and $^4I_{11/2}$ of Er$^{3+}$, and $^4I_{13/2}$ excited state, respectively.

The theoretical transmittance, $T$, of glass can be calculated as:

$$T = (1 - R)^2$$  \hspace{1cm} (2)

$$R = (n - 1)^2 / (n + 1)^2$$ \hspace{1cm} (3)

Where $R$ is reflection coefficient, and $n$ is the index of refraction. The theoretical transmittance calculated is 71.39%, which is close to the actual transmittance 75.46%. Thus, the glass has a good transmittance in the visible light and the near infrared band which is conducive to upconversion luminescence efficiency.

To understand the tolerance of the glass in an extreme environment, it is meaningful to study its optical characteristics after being exposed to radiation. After irradiation (The dose rate was 38 Gy min$^{-1}$, and the total dose of irradiation was 6840 Gy for 3 h), the transmittance dropped from 75.46% to 71.13%. It can be seen that
the radiation has little effect on the transmittance of the glass. This may be to the high atomic mass and large ray absorption cross section of heavy metal oxide glass [29], which makes the sample shows good radiation resistance.

3.3. The absorption and emission cross section

As illustrated in figure 4, the absorption cross section, $\sigma_{abs}(\lambda)$, is obtained by the absorption spectrum and the equation [30]:

$$\sigma_{abs}(\lambda) = \frac{2.303E(\lambda)}{N_d}$$  \hspace{1cm} (4)

Where, $\sigma_{abs}(\lambda)$ is the absorption cross section (cm$^2$), $E(\lambda)$ is the absorption spectrum value, $N$ is the number density of rare earth ions (cm$^{-3}$), $d$ is the thickness (mm).

According to the equation (4), the maximum absorption cross section is $1.370 \times 10^{-20}$ cm$^2$ at 525 nm.

The formula of integral absorption cross section of glass is [31]:

$$\sum \sigma_{abs}(\lambda) = \int \sigma_{abs}(\lambda) \, d\lambda$$  \hspace{1cm} (5)

The emission cross section of glass can be calculated by the following equation (6) [30, 32, 33]:

$$\sigma_{em}(\nu) = \sigma_{abs}(\nu) \exp \left\{ (\varepsilon - h\nu) / kT \right\}$$  \hspace{1cm} (6)

Where, $\sigma_{em}(\nu)$ is the emission cross section (cm$^2$), $\nu$ is light frequency, $k$ is the Boltzmann constant, $h$ is Planck’s constant, $\varepsilon$ is the net free energy excited from low to high levels at temperature $T$. Finally, the maximum stimulated emission cross section is $1.507 \times 10^{-20}$ cm$^2$ at 525 nm.

The relevant results are shown in the table 1.

3.4. Upconversion luminescence spectra

As shown in figure 5, the upconversion luminescence spectra of glass at 298, 323, 348, 373, 398, 423, 448, 473, and 498 K were measured under 980 nm laser excitation. As the temperature was increased, the upconversion luminescence intensity of the glass gradually decreased. According to the upconversion fluorescence spectra, the emission range of 518–540 nm is the transition of $^2H_{11/2} \rightarrow ^4I_{15/2}$ and the emission range of 540–570 nm is the transition of $^4S_{3/2} \rightarrow ^4I_{15/2}$. Under the quasi-thermal equilibrium, the number of ions on the $^2H_{11/2}$ and $^4S_{3/2}$ levels of the Er$^{3+}$ ion follows the Boltzmann distribution, and the fluorescence intensity ratio, $R$, of the thermally coupled level $^2H_{11/2} / ^4S_{3/2}$ can be expressed as [34, 35]:

![Figure 5. Upconversion luminescence patterns of $(La_{0.78}Er_{0.06}Yb_{0.16})(Ti_{0.95}Zr_{0.05})_{2.25}O_{6}$ glass at 298, 323, 348, 373, 398, 423, 448, 473, and 498 K.](image-url)
Figure 6. Fitting diagram of R and T of (La_{0.78}Er_{0.06}Yb_{0.16})Ti_{0.95}Zr_{0.05}O_{2.25} glasses.

Table 2. Fluorescence intensity ratio at 298, 323, 348, 373, 398, 423, 448, 473, 498 K of (La_{0.78}Er_{0.06}Yb_{0.16})Ti_{0.95}Zr_{0.05}O_{2.25} glasses.

| Temperature/K | $^3H_{11/2} \rightarrow ^4I_{15/2}$ | $^4S_{3/2} \rightarrow ^4I_{15/2}$ | R = $^3H_{11/2}/^4S_{3/2}$ |
|---------------|----------------------------------|----------------------------------|-----------------------------|
| 298           | 59948.40                         | 135952.22                        | 0.441                       |
| 323           | 61585.91                         | 122037.75                        | 0.505                       |
| 348           | 62096.43                         | 107156.97                        | 0.579                       |
| 373           | 68049.03                         | 105648.84                        | 0.644                       |
| 398           | 67562.62                         | 95241.87                         | 0.709                       |
| 423           | 66170.18                         | 86734.94                         | 0.763                       |
| 448           | 77576.96                         | 80938.00                         | 0.958                       |
| 473           | 73294.26                         | 69552.46                         | 1.054                       |
| 498           | 82168.24                         | 73614.14                         | 1.116                       |

Figure 7. The curves of relative sensitivity, S_r, of glass in the temperature range of 298–498 K.
Where, $I_{ij}$ is the $i$ energy level to $j$ level of fluorescence intensity, $W_{ij}$ is angular frequency of the radiation, $A_{ij}$ is $i$ level to $j$ energy level the probability of spontaneous emission transition, $g_i$ is I degeneracy of energy level, $\Delta E$ is the energy level difference, $k$ is the boltzmann constant, $T$ is the absolute temperature, $B$ is the correction factor.

Table 2 shows the relationship between fluorescence intensity ratio ($R$) and temperature ($T$).

According to equation (7), the fluorescence intensity ratio and temperature could be fitted, as shown in figure 6. The coefficients $A$ and $\Delta E/K$ are $11.64 \pm 4.02$ and $1308.06 \pm 200.80$ from the fitting results, respectively. By Calculating that the corresponding thermal coupling energy level difference $\Delta E$ is about $909.10$ cm$^{-1}$. The energy difference between $^2H_{11/2}$ and $^4S_{3/2}$ level is $\sim 780$ cm$^{-1}$. This deviation of $\Delta E$ from the
experimental data may be due to the overlap of $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ emission bands, and some potential non-radiative cross relaxation processes.

Sensitivity is the rate at which fluorescence intensity ratio changes with temperature. Moreover, it is the key parameter to evaluate temperature sensor. Relative sensitivity ($S_r$) can be expressed as [36]:

$$S_r = \frac{dR}{dT} = \frac{\Delta E}{kT^2}$$

According to equation (8), the relationship between relative sensitivity ($S_r$) and temperature is shown in figure 7. The obtained fitting equation is $S_r = 1308.6 / T^2$, the maximum relative sensitivity was calculated to be 1.47% K$^{-1}$ at 298 K. The value of relative sensitivity is bigger than Er$^{3+}$/Yb$^{3+}$ co-doped Sb$_2$O$_3$–WO$_3$–Li$_2$O glass-ceramic and TeO$_2$–ZnO–ZnF$_2$–La$_2$O$_3$ glass for optical temperature measurement [9, 16], indicating that the sample is a potential candidate material for optical temperature sensors.

### 3.5. Surface morphology

To discuss the mechanism of glass microcrystallization which can improve the upconversion luminescence [37], the surface structure of samples was studied. The selection of heat treatment temperature is from the temperature near crystallization temperature to the stage around the peak of crystallization to observe the change of crystal structure. Therefore, the heat treatment temperature range of 875 °C–925 °C was selected in paper.

To analyze the crystallization behavior of glass during heat treatment, the glass before and after heat treatment was analyzed by XRD, as shown in figure 8. Obviously, there is no sharp diffraction peak in the diffraction pattern of unheated glass sample. After heat treatment, there are several very clear sharp peaks, which indicates that crystals have been precipitated in the glass.

Figure 9 shows the SEM of glass at different heat treatment temperatures. Figure 9 (a) shows the surface morphology of glass without heat treatment, and its surface is smooth. Figures 9(b)–(d) shows the surface morphologies of samples under heat treatment at 875 °C, 900 °C and 925 °C for 50 min, respectively. Several crystal particles were observed gradually growing on the glass matrix. A large number of microcrystals of
different sizes and morphologies appeared on the glass surface. The crystal structure is dense and can provide strong lattice coordination field, which can reduce the interaction of rare earth ions and obtain excellent upconversion luminescence performance. Therefore, the presence of microcrystals is expected to improve the upconversion luminescence intensity.

To further investigate the grain growth in glass-ceramics, TEM and SAED were performed for glass-ceramics, as shown in figure 10. Figure 10 shows the formation of columnar crystals with a diameter of ~100 nm and different lengths. Figure 10(a) shows the sample heat treated at 875 °C, and the sharp spots in SAED spectrum show that the grains have good monoclinicinity. Figures 10(b), (c) shows the samples heat treated at 900 °C and 925 °C, respectively, although they have the formation of columnar crystals with a diameter of ~100 nm, the SAED spectrum is disordered. Therefore, the crystal in figures 10(b), (c) is not the best environment for rare earth ions to obtain strong upconversion luminescence. The single crystal obtained by heat treatment at 875 °C is conducive to improving the luminescence intensity and can offer an excellent matrix material for rare earth ion luminescence.

3.6. Acid and alkali resistance
To evaluate the chemical stability of the glass, HCl (36–38 wt%) and NaOH (33 wt%) solution were used to etch the obtained samples at 200 °C for 50 h, respectively. Two samples were used and weighed the etched glass three times to take its average value.

Figure 11 shows the relative mass loss of the etched glass in acid and alkali solution. The relative mass loss first increased and then decreased. The maximum relative mass loss of glass was 0.80 × 10⁻² mg after etching for 25 h, indicating that the glass has good acid and alkali resistance. In addition, the relative mass loss of the etched glass fluctuates, which may be due to a small amount of etching liquid remaining inside or fluctuations in the balance.

Figure 12 shows the SEM images of the etched glass, with some etched pits present on the glass surface. Figure 12(a) shows the SEM morphology of acid etched glass. The chemical stability of glass is largely determined by the tightness of the glass phase network structure. Therefore, it can be hypothesized that the acid resistance of the glass gradually deteriorates due to the diffusion of rare earth ions from the glass network structure, which causes the etched pits. Figure 12(b) shows some erosion pits on the surface of glass, this may be due to the higher activation energy required in the glass dissolution process. Finally, according to the SEM results, it is concluded that the alkali resistance of the glass is better than the acid resistance of the glass.

4. Conclusions
In conclusion, (La₀.₇₈Er₀.₀₆Yb₀.₁₆)(Ti₀.₉₅Zr₀.₀₅)₂.₂₅O₆ glasses were successfully fabricated by aerodynamic levitation methods. The density of the glass was 5.346 g cm⁻³. The glasses show high refractive index of 2.30 and Abbe number of 18.57, indicating that the glass has good optical performance. Before irradiation, the maximum transmittance of the glass from visible to near infrared band reached 75.46%. The maximum transmittance of irradiated glass from visible to near-infrared band is 71.13%, indicating that the irradiation has little negative effect on the transmittance of glass. The maximum absorption and maximum emission cross section are 1.370 × 10⁻²⁰ cm² and 1.507 × 10⁻²⁰ cm² at 525 nm, respectively. The microstructures of the samples were analyzed by XRD, SEM and TEM. The XRD patterns confirmed the crystallization behavior of glass during heat treatment. SEM images of glass-ceramics heated at different temperatures present that the surface of glass-ceramics has crystal particles. TEM results show that the samples heat treated at 875 °C for 50 min have best
single crystallinity, which is helpful to improve the luminescence intensity of rare earth ions. The results provide a reference for the further study of rare earth ions and matrix materials to improve the upconversion luminescence properties. More importantly, the optical thermometry behavior has been studied between its upconversion green emissions, along with high relative sensitivity of 1.47% K⁻¹ at 298 K. Results indicate that Er³⁺/Yb³⁺ co-doped TiO₂–ZrO₂–La₂O₃ glasses is an excellent temperature-sensing material with high relative sensitivity. The acid and alkali resistance experiment shows that the alkali resistance of the glass is better than that of the acid resistance. The research results have reference value for the tolerance research of heavy metal oxides in extreme environment and the field of temperature sensing.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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