



Emission efficiency at 1 μm from low Yb^{3+} concentrated tellurite glass-ceramics: Alternative materials for the future rare-earth metal shortage

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ABSTRACT

Glasses in the $\text{TeO}_2\text{-ZnO-Bi}_2\text{O}_3$ system were prepared with up to 2.5 mol% of Yb_2O_3 using standard melting process and their crystallization process was investigated for the first time. No concentration quenching was observed. The thermal treatment leads to surface precipitation of crystals, the composition of which depends on the Yb_2O_3 content. The addition of Yb_2O_3 in the tellurite network promotes the precipitation of $\text{Bi}_2\text{Te}_4\text{O}_{11}$ crystals at the expense of $\text{Zn}_2\text{Te}_3\text{O}_8$ crystals. The growing of these crystals in the low Yb^{3+} concentrated glass during a thermal treatment increases the interaction between the Yb^{3+} ions leading to an enhancement of the Yb^{3+} emission properties which reach those of highly Yb^{3+} concentrated tellurite glass. Our study suggests that a thermal treatment can be a practical alternative to increase the emission efficiency of the glass prepared with 0.5 mol% of Yb_2O_3 to the level of the as-prepared glass doped with 2.5 mol% of Yb_2O_3 .

Rare-earth (RE) ions have quickly grown interest due to advances in electronics and other technologies, leading to a significant increase in their consumption over the past decades. Among the RE ions, Yb^{3+} has been of great interest due to its simple energy level scheme which is composed of a single optically excited level ($^2\text{F}_{5/2}$). Yb^{3+} is an important RE for various photonics applications such as infrared lasers and amplifiers with wavelength near 1 μm for example [1]. As a glass host for RE ions, tellurite glasses are known to have wide transmission window, low phonon energy, and good optical, chemical, and mechanical properties [2]. Compared to silica glass, tellurite glasses have higher refractive index (up to 2.2) and therefore higher absorption and stimulated emission cross sections. Additionally, they can incorporate a larger amount of RE than silica glass; the formation of RE clusters occurs at higher RE doping levels than in silica glass. Therefore, tellurite glasses are ideal hosts for Yb^{3+} ions [3]. Although TeO_2 is a glass former, it cannot form a glass on its own. Therefore, there has been an interest in developing new tellurite glasses with a large variety of chemical compositions to change their network structure [3] and so their spectroscopic properties [4–8]. It is crucial that these newly developed tellurite glasses should incorporate large amount of Yb^{3+} for high performance before the emission lifetime and intensities begin to be affected by RE concentration quenching [9,10]. Indeed, low Yb_2O_3 content often leads

to a small absorption coefficient.

However, one should not forget that RE ions are economically valuable yet exposed to inventory shortage. Actually, as most of the global supply of RE is produced in China, there is an uncertainty regarding the stability of supply [11]. Despite RE supply chains in Canada, Australia, Russia, and in the Pacific Ocean, the price of RE materials keeps increasing due to global supply restrictions recently. The other issues with RE is related to the difficulty in separating them from each other during recycling to harvest pure single elements [12]. Therefore, there is a need for alternative solutions than increasing the RE content to enhance the spectroscopic properties of new optical materials for photonics.

One method to enhance the spectroscopic properties of a RE doped glass is to perform a thermal treatment to the glass to induce the nucleation and growth of crystals. Such materials are known as glass-ceramics (GC), defined by Stookey as “Glass-ceramics are made by first melting and forming special glasses containing nucleating agents and then causing controlled crystallization of the glass” [13]. GCs possess improved properties such as mechanical and thermal properties compared to parent glass. For the GCs to be promising as luminescent materials, the crystals should contain the RE and should have a specific crystalline phase in order to obtain an enhancement of the GCs

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spectroscopic properties.

Tellurite glasses within the $\text{TeO}_2\text{-ZnO-Bi}_2\text{O}_3$ system are promising glasses for the development of glass-ceramic as zinc- and bismuth-based crystals were reported to precipitate in the volume of the glass during a thermal treatment [14]. The addition of Er^{3+} in the glass network was reported to change the crystallization process to surface crystallization [15]. The thermal treatment led to the formation of $\text{Bi}_{3.2}\text{Te}_{0.8}\text{O}_{6.4}$ crystals which was reported to decrease the distance between the Er^{3+} ions enhancing the glass spectroscopic properties clearly demonstrating the potential of this glass composition for photonics applications [15]. While numerous studies have been focused on the formation of Yb^{3+} , Er^{3+} codoped glass-ceramics, there are comparatively fewer studies on Yb^{3+} single-doped glass-ceramics, particularly on Yb^{3+} doped tellurite glass-ceramics.

In this paper, the crystallization process of Yb^{3+} tellurite glasses prepared with different amount of Yb_2O_3 is reported for the first time. The spectroscopic properties of new tellurite glasses prior to and after thermal treatment are reported in order to investigate the impact of the crystallization on the Yb^{3+} spectroscopic properties.

Glasses with the composition $(100 - x)(0.70 \text{ TeO}_2 - 0.20 \text{ ZnO} - 0.10 \text{ Bi}_2\text{O}_3) - x \text{ Yb}_2\text{O}_3$ (in mol%) with $x = 0.5, 1, 1.25$ and 2.5 (labeled as Yb0.5, Yb1, Yb1.25 and Yb2.5 respectively) were prepared using standard melting process in normal atmosphere. The precursors were TeO_2 (Thermo Fisher, >99.9 %), Bi_2O_3 (Thermo Fisher, 99.9 %), ZnO (Sigma-Aldrich, 99.9 %) and Yb_2O_3 (Thermo Fisher, 99.99 %). The 10g batches were melted in quartz crucible for 40 min at 750°C (Yb0.5 and Yb1) and at 775°C (Yb1.25 and Yb2.5) and, after quenching, annealed at $\sim 40^\circ\text{C}$ below the glass transition temperature (T_g) of the glasses for 4 h to release the internal stress from the quench. Finally, the glasses were heat treated at ($T_g + 20^\circ\text{C}$) for 17 h and then at 430°C or 450°C for 1 h.

A Scanning Electron Microscope (SEM, Crossbeam 540 Carl Zeiss) with Energy Dispersive Spectroscopy (EDS) detector (X-MaxN 80, Oxford Instruments, Abingdon-on-Thames, UK) was used to analyze the glasses after the thermal treatment.

The glass density, with an accuracy of $\pm 0.02 \text{ g/cm}^3$, was measured by the Archimedes method, using ethanol as immersion liquid.

X-Ray Diffractometer (XRD) analyzer (PANalytical Empyrean) with $\text{Cu K}\alpha$ X-ray radiation ($\lambda = 1.54 \text{ \AA}$) was used to identify the crystalline phases precipitating in the glasses during thermal treatment. Data are collected from $2\theta = 20$ to 60° with a step size of 0.05° .

The thermal properties of the glasses were obtained with an accuracy of $\pm 3^\circ\text{C}$, using differential thermal analysis (DTA, Netzsch JUPITER F1 instrument). The measurements were carried out in a Pt crucible using a heating rate of $10^\circ\text{C}/\text{min}$. The T_g was taken at the inflection point of the endothermic peak obtained by taking the first derivative of the DTA curve, T_p at the maximum of the exothermic peak and T_x at the onset of the crystallization peak. The difference between T_x and T_g (ΔT) provides information about the thermal stability of the glass against crystallization.

The Raman spectra were measured with a Renishaw inVia Qontor Raman microscope equipped with a cooled charge coupled device (CCD) camera using a 785 nm laser for the excitation and a 1200 lines/mm grating.

The absorption spectra were obtained using UV-Vis-NIR spectrophotometer (UV-3600 Plus, Shimadzu). The absorption cross-section

$\sigma_{\text{abs}}(\lambda)$ was calculated from absorbance as in [7,15]. The accuracy of the absorption cross-section is given at $\pm 10\%$.

The emission spectra of the glasses were collected from the glasses crushed into powder at room temperature using a continuous-wave monochromatic single-mode fiber pigtailed laser diode (CM962UF76P-10R, Oclaro) temperature tuned to 960 nm for excitation.

Yb^{3+} doped tellurite glasses within the $\text{TeO}_2\text{-ZnO-Bi}_2\text{O}_3$ system were prepared with different Yb_2O_3 contents, 2.5 mol\% being the maximum amount of Yb_2O_3 which could be incorporated into the tellurite glass matrix without inducing crystallization during the quenching step. The physical and thermal properties of the investigated glasses are listed in Table 1. As expected, an increase in x leads to a slight increase in the density due to addition of the Yb_2O_3 with high molar mass in the glass network. An increase in x has a noticeable impact on the glass thermogram as seen in Fig. 1a. An increase in T_g with an increase in x is observed suggesting a strengthening of the tellurite network when adding Yb_2O_3 . Additionally, two maxima are suspected in the exothermic peak, the distance of which increases as x increases suggesting that the addition of Yb_2O_3 in the tellurite network has an impact on the crystallization mechanism as suggested by Lemiere et al when adding Er^{3+} in the same glass network [15]. The large ΔT of the glasses suggest their good stability against crystallization.

The normalized Raman spectra of the glasses, presented in Fig. 1b, are typical for tellurite glasses. They exhibit 3 bands at $400\text{-}500 \text{ cm}^{-1}$, 664 cm^{-1} and 745 cm^{-1} . These bands can be related to Te-O-Te/O-Te-O symmetrical bending and stretching modes found at corner sharing sites of TeO_4 , TeO_3 and TeO_{3+1} polyhedra [16], to the symmetrical stretching vibrational modes of TeO_4 trigonal bipyramid units, more specifically to the antisymmetric vibrations of Te-O-Te linkages between TeO_4 units [17,18] and to the symmetrical stretching of Te-O and Te=O bonds which contain non-bridging oxygens (NBOs) in TeO_3 trigonal pyramid units and TeO_{3+1} polyhedra [16,19], respectively. The shoulder at $\sim 440 \text{ cm}^{-1}$ can be also associated with the symmetric stretching vibrations mode of Zn-O in ZnO_4 unit [20]. A slight decrease in intensity of the bands in the $400\text{-}500 \text{ cm}^{-1}$ range and at 664 cm^{-1} compared to that of the band at 745 cm^{-1} can be seen with an increase in x . These changes suggest that some depolymerization of the glass network associated with a slight decrease in the amount of TeO_4 units at the expense of TeO_3 and TeO_{3+1} units is suspected to occur when adding Yb_2O_3 in the tellurite glass. Elkhoshkhany et al reported similar changes in the structure when adding Yb_2O_3 into tellurite network and related them to the breakage of the $(\text{Te}_{\text{ax}}\text{-O})$ bonds of TeO_4 by Yb_2O_3 leading to the formation of TeO_3 and TeO_{3+1} units [21].

The absorption spectra of the glasses are presented Fig. 1c. A small shift of the optical band gap to higher wavelength is suspected with the addition of Yb_2O_3 due to the formation of NBOs as discussed earlier. The absorption spectra also exhibit the typical absorption band of Yb^{3+} ions which corresponds to the $4f\text{-}4f$ transitions of Yb^{3+} from the ground state ($^2F_{7/2}$) to the excited state ($^2F_{5/2}$) (Fig. 1d). As expected, the larger x , the larger absorption coefficient. As seen in Table 1, all glasses possess similar absorption cross-section at 975 nm within 10% which is higher than the one reported for commercial Kigre QX/Yb ($1.06 \times 10^{-20} \text{ cm}^2$) laser glass but in a good agreement with the absorption cross-section reported for other Yb^{3+} doped tellurite glasses [6,7].

The glasses exhibit also the typical emission band from Yb^{3+} ions

Table 1

Physical and thermal properties of the investigated glasses, their absorption and coefficient and cross-section and their emission cross-section at 975 nm .

x	ρ ($\text{g}\cdot\text{cm}^{-3}$) $\pm 0.02 \text{ g}\cdot\text{cm}^{-3}$	T_g ($^\circ\text{C}$) $\pm 3^\circ\text{C}$	T_x ($^\circ\text{C}$) $\pm 3^\circ\text{C}$	$T_{p1}\text{-}T_{p2}$ ($^\circ\text{C}$) $\pm 3^\circ\text{C}$	$\Delta T=T_x\text{-}T_g$ ($^\circ\text{C}$) $\pm 6^\circ\text{C}$	Yb^{3+} (ions $\cdot\text{cm}^{-3}$) (10^{20}) $\pm 5\%$	α_{abs} (cm^{-1}) at 975 nm	σ_{abs} at 975 nm (10^{-20}cm^2) $\pm 10\%$
Yb0.5	6.17	337	423	496-510	86	2.1	3.27	1.54
Yb1	6.19	340	429	483-513	89	4.2	5.94	1.41
Yb1.25	6.19	340	434	488-513	94	5.3	6.73	1.28
Yb2.5	6.21	354	440	457-503	86	10.4	13.88	1.34

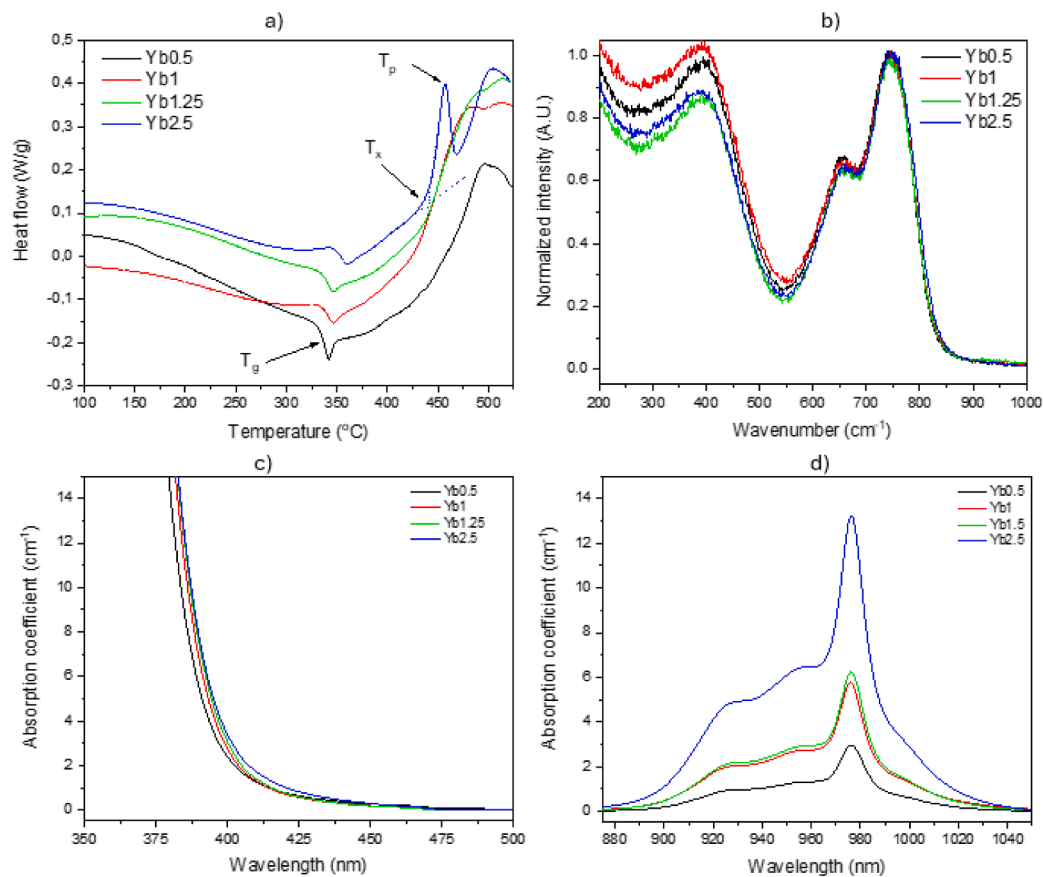


Fig. 1. Thermograms (a), normalized Raman spectra (b), absorption spectra (c) and Yb³⁺ absorption band (d) of the investigated glasses.

when located in an amorphous network (Fig. 2a). This band is due to the $^2F_{5/2} \rightarrow ^2F_{7/2}$ transition of Yb³⁺. The shape of the emission depends on the Yb₂O₃ content. Not only an increase in x leads to a decrease in intensity of the band at 975 nm compared to the signal at 1000 nm and to an increase of the bandwidth (Table 2), but it increases also the area of the emission band (Fig. 2b). According to [22,23], these changes in the Yb³⁺ spectroscopic properties with the increase in Yb₂O₃ content could be related to the effective radiation trapping due to its close overlap with the absorption peak wavelength. An unexcited Yb³⁺ ions is suspected to reabsorb the emitted photons which is then trapped. The large bandwidth of the emission band of the Yb2.5 is caused by the increase in

Table 2
Bandwidth of the emission band measured using a 960 nm excitation.

X	As-prepared glasses (nm)	After ($T_g + 20^\circ\text{C}$) for 17 h - 430 °C for 1 h (nm)	After ($T_g + 20^\circ\text{C}$) for 17 h - 450 °C for 1 h (nm)
Yb0.5	72	72	70
Yb1	71	71	73
Yb1.25	70	74	75
Yb2.5	84	83	86

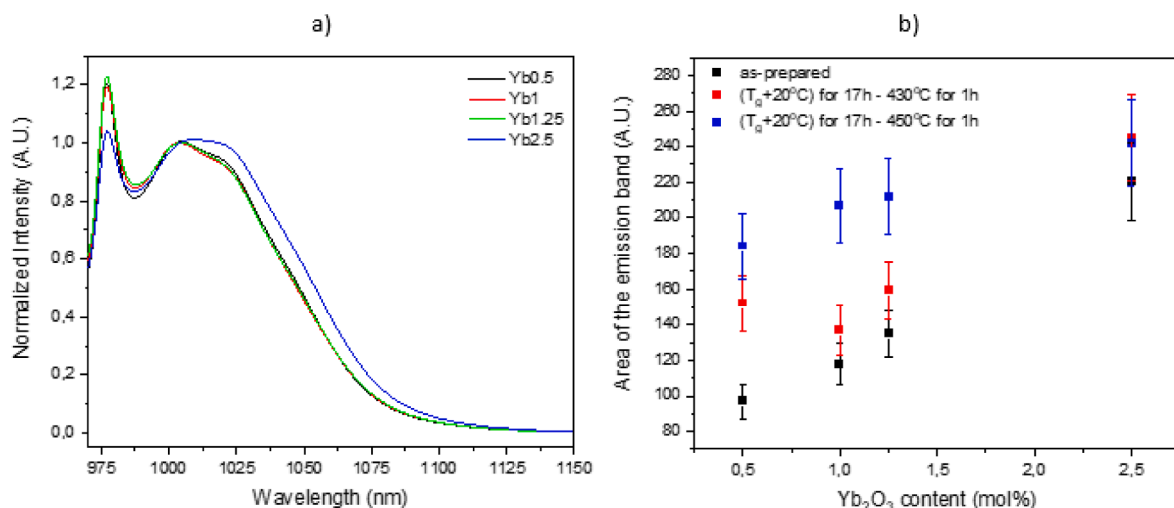


Fig. 2. Normalized emission spectra of the glasses ($\lambda_{exc} = 960$ nm) (a) and area of emission band of the glasses prior to and after heat treatment (b).

intensity of the shoulder at 1050 nm and could be related to the energy migration among Yb^{3+} excitons induced by the decrease in the inter ionic distance when the concentration of Yb_2O_3 reaches 2.5 mol%. Nonetheless, it is clear from Fig. 2b that no concentration quenching occurs when adding up to 2.5 mol% of Yb_2O_3 in the tellurite glass.

The glasses were heat-treated at their respective ($T_g + 20^\circ\text{C}$) for 17 h and then at 430°C or 450°C for 1 h. After the thermal treatment, the glasses were opaque, a clear sign of crystallization. As reported for the Er^{3+} doped tellurite glasses, surface crystallization was observed. The XRD patterns of the heat-treated glasses (Fig. 3a) clearly evidence the appearance of sharp peaks after the thermal treatment confirming the precipitation of crystals occurring during the heat treatment. The XRD peaks seen in the XRD pattern of Yb0.5 can be related to $\text{Bi}_2\text{Te}_4\text{O}_{11}$ [00-026-0227] and $\text{Zn}_2\text{Te}_3\text{O}_8$ [00-044-0241], similarly to the crystals found in the heat-treated Er^{3+} doped tellurite glass within the same glass system [15]. An increase in the growth temperature from 430 to 450°C increases the intensity of the peaks related to the $\text{Bi}_2\text{Te}_4\text{O}_{11}$ as compared to the peaks related to $\text{Zn}_2\text{Te}_3\text{O}_8$. An increase in the Yb_2O_3 content leads to a decrease in intensity of the peak related to the $\text{Zn}_2\text{Te}_3\text{O}_8$ crystals and also to the appearance of new crystals which are thought to be related to $\text{Yb}_2\text{Te}_5\text{O}_{13}$ [04-012-2916]. The analysis of the XRD patterns is confirmed by the SEM images and the elemental mapping taken from the cross section of the heat-treated glasses (Fig. 3b): the addition of 2.5 mol % of Yb_2O_3 in the tellurite network is suspected to promote the

precipitation of $\text{Bi}_2\text{Te}_4\text{O}_{11}$ crystal at the expense of $\text{Zn}_2\text{Te}_3\text{O}_8$. Precipitation of Yb containing crystals is also expected from the elemental mapping of Yb which shows Yb rich area in the heat-treated Yb2.5 glass.

Except for the Yb2.5 glass, the precipitation of the crystals in the glasses has an impact on their Yb^{3+} spectroscopic properties: not only the thermal treatment increases the intensity of the Yb^{3+} emission (Fig. 2a), but it also modifies the shape of the emission band (Fig. 4) and so the emission bandwidth (Table 2). As the emission band remains broad after the thermal treatment, the Yb^{3+} ions are assumed to remain in the amorphous part of the heat-treated glass. The intensity of the band at 975 nm decreases as compared to the band at 1000 nm suggesting that the precipitation of the crystals in the tellurite network decreases the Yb-Yb inter ionic distance allowing the energy migration among Yb^{3+} excitons. The position of the zero-phonon line at 975 nm shifts to longer wavelength after the thermal treatment indicating changes in the crystal field strength due to modification in the $^2\text{F}_{7/2}$ fundamental multiplet splitting as explained in [7]. As the emission properties of the Yb2.5 glass are not modified after the thermal treatment, it is possible to think that the Yb^{3+} sites and the Yb-Yb distance in a depolymerized network are not impacted by the precipitation of the crystals. One should mention that the amount of the $\text{Yb}_2\text{Te}_5\text{O}_{13}$ crystals is probably too small to impact the spectroscopic properties of the heat-treated Yb2.5 glass.

It is interesting point out that the heat-treated glasses exhibit similar emission intensity and emission band than the as-prepared Yb2.5 glass

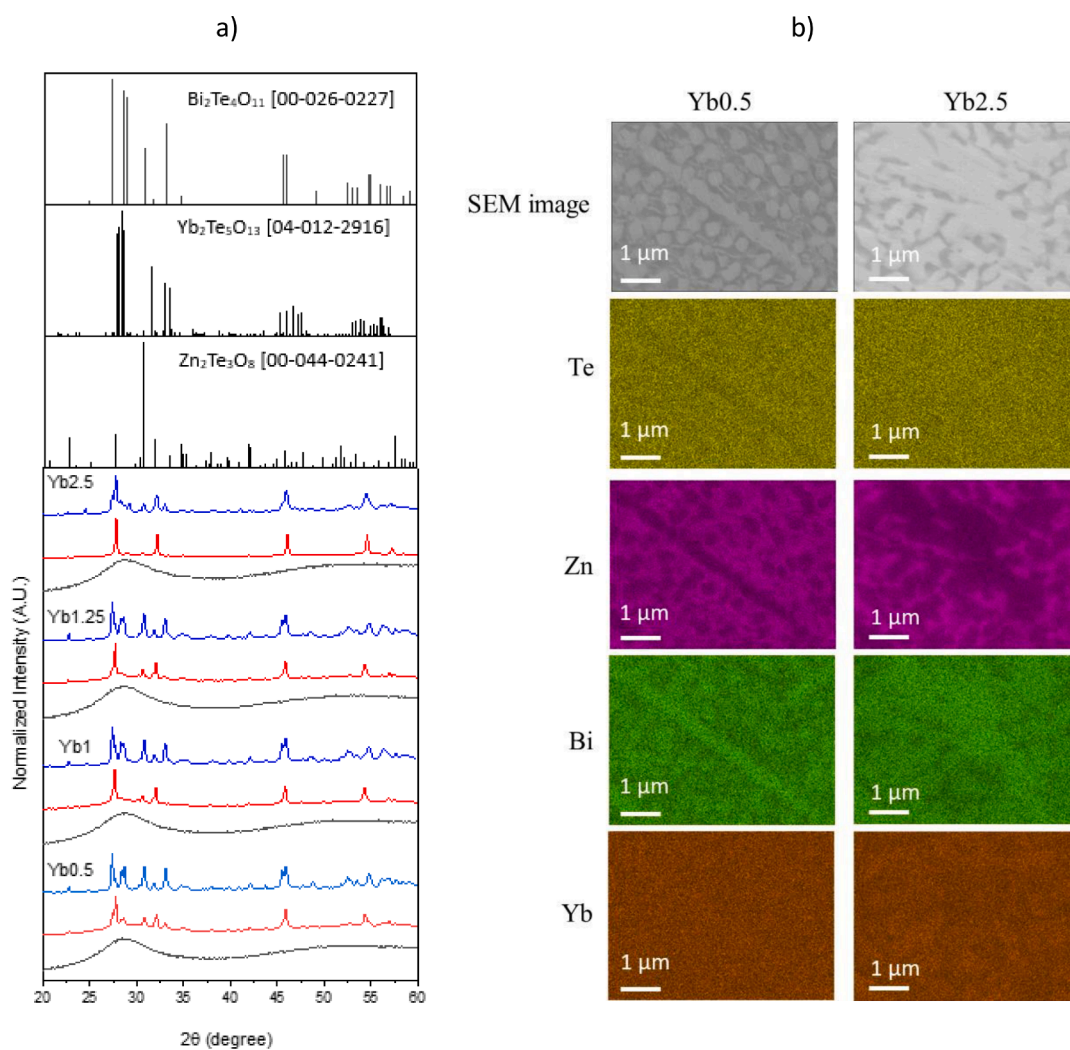


Fig. 3. X-ray diffractometer (XRD) pattern of the as-prepared glasses (black), of the glasses after heat treatment at ($T_g + 20^\circ\text{C}$) for 17 h and at 430°C for 1 h (red) and at ($T_g + 20^\circ\text{C}$) for 17 h and at 450°C for 1 h (blue). Also shown are the XRD pattern of the expected crystals (a). SEM image of the cross-section of the Yb0.5 and Yb2.5 glasses heat treated at ($T_g + 20^\circ\text{C}$) for 17 h and at 450°C for 1 h and the corresponding elemental mapping.

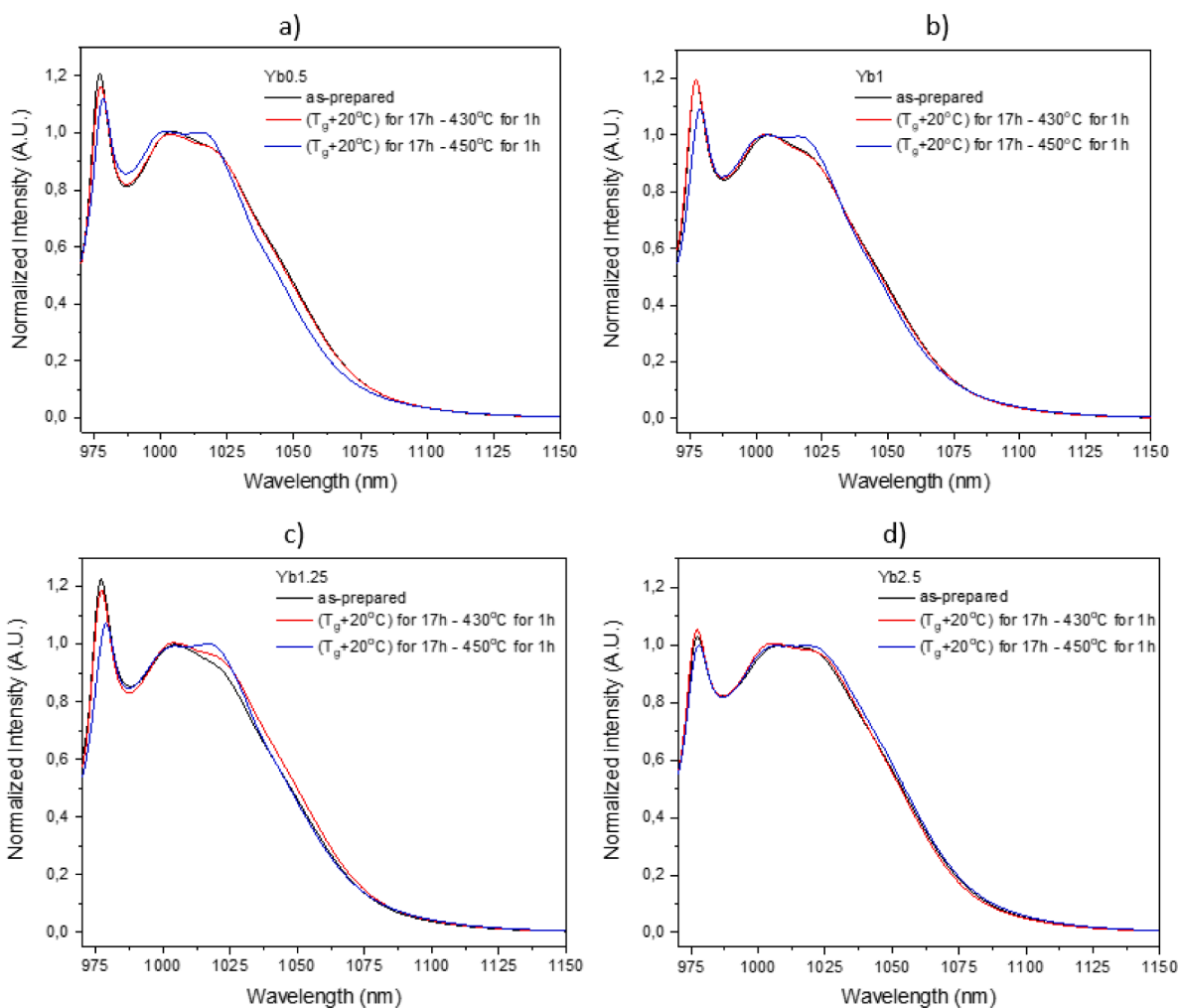


Fig. 4. Normalized emission spectra of the Yb0.5 (a); Yb1 (b), Yb1.25 (c) and Yb2.5 (d) glasses prior to and after heat treatment ($\lambda_{exc} = 960$ nm).

suggesting that the precipitation of the crystals in the low Yb_2O_3 concentrated glasses leads to similar Yb^{3+} sites to those found in the highly Yb_2O_3 concentrated glasses.

In summary, novel Yb^{3+} doped tellurite glasses were prepared using standard melting process and their crystallization process was investigated. An increase in the Yb_2O_3 content leads to an increase the intensity of the emission at $\sim 1 \mu\text{m}$ and to a slight depolymerization of the tellurite network which modifies the nucleation mechanism of the glass. We showed for the first time that the precipitation of crystals in low Yb^{3+} concentrated glasses in the TeO_2 - ZnO - Bi_2O_3 network can be used to increase the intensity of the Yb^{3+} emission to the level of highly Yb^{3+} concentrated glasses.

CRediT authorship contribution statement

M. Bardins: Writing – review & editing, Methodology. **N. Vakula:** Writing – review & editing, Validation, Supervision, Methodology, Formal analysis, Conceptualization. **L. Petit:** Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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