Glasses formation, characterization, and crystal-structure determination in the Bi$_2$O$_3$–Sb$_2$O$_3$–TeO$_2$ system prepared in an air

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Abstract A glass-forming domain is found and studied within Bi$_2$O$_3$–Sb$_2$O$_3$–TeO$_2$ system. The glasses composition were obtained in pseudo-binary xSbO$_{1.5}$, (1–x)TeO$_2$ for 0.05 ≤ x ≤ 0.20. The constitution of glasses in the system Sb$_2$O$_3$–TeO$_2$ was investigated by DSC, Raman, and Infrared spectroscopy. The influence of a gradual addition of the modifier oxides on the coordination geometry of tellurium atoms has been elucidated based Infrared and Raman studies and showed the transition of TeO$_4$, TeO$_3$ and TeO$_3$ units with increasing Sb$_2$O$_3$ content. XRD results reveal the presence of three crystalline: γ-TeO$_2$, α-TeO$_2$, and SbTe$_3$O$_8$ phases during the crystallization process. The density of glasses has been measured. The investigation in the ternary system by the solid state reaction using XRD reveals the existence of a solid solution Bi$_{1-x}$Sb$_1-x$Te$_2$O$_4$ isotopic to BiSbO$_4$ with 0 ≤ x ≤ 0.1.

Introduction

Tellurium dioxide (TeO$_2$) is an important material in both amorphous as well as crystalline form and finds application in active optical devices in particular, a huge hyper-susceptibility, deflectors, modulators, γ-ray detectors, and gas sensors because of its high acousto-optic figure of merit, chemical stability, and mechanical durability [1–6]. It is also not hygroscopic and has superior physical properties such as high dielectric constant and low melting point (800 °C) [7–10]. The origin of the extraordinary non-linear optical properties of TeO$_2$-based glasses is attributed to high hyperpolarizability of a lone electron pair related to the 5 s orbital of tellurium atom. Presently, the well-recognized three modifications of crystalline TeO$_2$ are α-TeO$_2$, β-TeO$_2$, and γ-TeO$_2$ [11–16]. Of these, recently documented γ-TeO$_2$ phase has gained a lot of attention for nonlinear optical designs and efforts are made to understand its properties in bulk crystal and glass using as Fourier transform infra-red spectroscopy (FTIR) and Raman spectroscopy. It has been reported that γ-TeO$_2$ phase appears as the first crystalline structure during the temperature-induced crystallization of TeO$_2$ glass [17–20]. TeO$_2$ glass is not stable. An addition of second oxide component M$_n$O$_m$ makes glasses structures more stable [21–28].

In the present study, we have revisited this system in one hand to obtain more information on the structure of these glasses reported by Charton et al. in Sb$_2$O$_4$–TeO$_4$ system [29, 30]. On the other hand we report the formation, the thermal properties and the local structure using Infrared and Raman studies of glasses prepared in the TeO$_2$–Sb$_2$O$_3$ pseudo-binary and crystalline phases in ternary system. A detailed analysis of the crystalline phase formation in this system synthesized in an oxygen flow will be described successively also.
Experimental procedure

The amorphous and crystalline samples were prepared using high purity commercial materials Bi₂O₃, TeO₂, Sb₂O₃ of analytical grade (Aldrich 99.9%). The batches of suitable proportions of starting products were mixed in an agate mortar and then heated in air at 800 °C (20 min) for vitreous phases and at 600–800 °C (48 h) for crystalline phases. All of them are quenched to room temperature and identified by X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer (Cu-Kα radiation). \( T_g \) (glass temperature) and \( T_c \) (crystallization temperature) were determined using Differential Scanning Calorimetry (DSC) Netsch 2000 PC type from powder samples glasses for about 8 mg in aluminum pans. A heating rate of 10 °C/min was used in the 30–600 °C range. Infrared absorption measurements between 2,000 and 400 cm⁻¹ were made for powder specimens dispersed in a pressed KBr disk. The Raman spectra were recorded in the 80–1,000 cm⁻¹ range using high purity commercial materials Bi₂O₃, TeO₂, Sb₂O₃.

Glass composition, their respective thermal parameter, density and molar volume

Table 1 Glass composition, their respective thermal parameter, density and molar volume

| % mol TeO₂ | % mol SbO₁,₅ | \( T_g \) (°C) | \( T_{c₁} \) (°C) | \( T_{c₂} \) (°C) | \( T_{c₃} \) (°C) | \( T_{c₃} - T_g \) | Density \( (±0.02) \) g/cm³ | Molar volume (cm³/mol) |
|------------|--------------|----------------|----------------|----------------|----------------|-----------------|-----------------|-----------------|
| 95         | 5            | 328            | 405            | 497            | 573            | 77              | 5.95            | 27.93           |
| 90         | 10           | 329            | 409            | 519            | 571            | 80              | 6.00            | 28.80           |
| 85         | 15           | 334            | 430            | 509            | 575            | 96              | 6.10            | 29.40           |
| 80         | 20           | 339            | 437            | 500            | 563            | 98              | 6.18            | 30.09           |

![Fig. 1 Phase diagram of Bi₂O₃–Sb\(^{1+3,5}\)O₂–TeO₂ system (colored area = vitreous domain) to the one proposed by Charton et al. in the \( xSb_2O_3 \), \( 1-x \) TeO₂ system study where 0.02 \( \leq x \leq 0.175 \) [29, 30].](Image 344x588 to 490x715)

Result and discussion

A wide range glass system based on the Bi₂O₃–Sb₂O₃–TeO₂ system was prepared at 800 °C. This temperature has been chosen to have a homogenous reagent in one hand and to avoid volatilization of TeO₂ at high temperature \( (T_{TeO₂, Melting} = 732 \) °C) on the other hand (Fig. 1). The color of the glass changes slightly from dark yellow to yellow with increasing Sb₂O₃ and Bi₂O₃ concentration. The glassy domain obtained interval in the pseudo-binary \( xSbO₁,₅ \), \( (1-x)TeO₂ \) 0.05 \( \leq x \leq 0.20 \) is slightly different compared to the one proposed by Charton et al. in the \( xSb_2O_3 \), \( 1-x \) TeO₂ system study where 0.02 \( \leq x \leq 0.175 \) [29, 30].

Differential scanning calorimetry

Series of glasses composition are listed in Table 1. An addition of SbO₁,₅ (up to 5 mol%) would result in the increase of glass stability (as indicated by \( T_g - T_c \)). This is presumably due to the participation of Sb\(^{1+3/2}\) in the glass network. The values of \( T_g, T_{c₁}, T_{c₂}, \) and \( T_{c₃} \) are presented (Fig. 2 and Table 1).

The DSC curves exhibit an endothermic effect due to glass transition (\( T_g \)). At higher temperatures three exothermic peaks are observed and related to temperature crystalline phases. Figure 2 shows the dependence of characteristic temperature, glass transition, the first crystallization (\( T_{c₁} \)), the second (\( T_{c₂} \)), and the third crystallization (\( T_{c₃} \)) on Sb₂O₃ content. The appearance of single peak (all glasses) due the glass transition temperature indicates the homogeneity of the glasses prepared. With increasing in the concentration of Sb₂O₃ in the glass matrix, the \( T_g \) increases and the difference (\( T_c - T_g \)) (about 77–98 °C) implies a thermal stability of glasses. In a study of alkali tellurite glasses, Pye et al. [31] showed that the temperature of the glass transition decreases with increasing amount of Li, Na, or K compound. The dependence of Sb₂O₃ content shows a different tendency...
especially of glass transition compared with the alkali tellurite glasses. The alkali atoms easily move in the glass structure. However, antimony atoms move with greater difficulty in the glass, because the Sb atom is restrained by relatively strong bonds to every coordinate oxygen. The slight change of the temperature of crystallization of a vitreous composition to another is due to the kinetic phenomenon. Based on XRD and DSC analysis for glassy samples 5–20 mol% SbO\(_{1.5}\) (see Fig. 3) a first peak of crystallization corresponds to the \(\gamma\)TeO\(_2\), \(\alpha\)TeO\(_2\), and SbTe\(_3\)O\(_8\) at 405–437 °C range. This phenomenon which we observed the crystallization \(\gamma\)TeO\(_2\) variety could be expected: similar behavior has been observed in the many others systems as TeO\(_2\)–WO\(_3\) [15], Nb\(_2\)O\(_5\)–TeO\(_2\) [13, 16], TeO\(_2\)–ZnO [18] and TeO\(_2\)–SrO [19]. In second crystallization ranging 497–519 °C belongs to reinforcing SbTe\(_3\)O\(_8\) and TeO\(_2\)\(\alpha\) phases. The last peak (563–573 °C) with weak intensity is attributed to totally transformation \(\gamma\)TeO\(_2\) metastable polymorph into the stable \(\alpha\)TeO\(_2\) and SbTe\(_3\)O\(_8\). It can be observed that there is a linear relationship between \(T_c - T_g\) and \(T_c\) against the Sb\(_2\)O\(_3\) content. This indicates that the glass is easily fabricated. The increase in glass stability is also reported to be due to the structural formation of SbTe\(_3\)O\(_8\) units.

Density

The density of the specimens was measured using Archimeds principle using orthophalate as the immersion liquid (\(d_{\text{orthophalate}} = 1.11712\) at 22 °C). A glass disc was weighted in air (\(W_{\text{air}}\)) and immersed in orthophalate and reweighted (\(W_{\text{orthophalate}}\)). The relative density is given by the following relation [22]:

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**Fig. 2** DSC curves of glassy samples obtained in \(1-x\)TeO\(_2\), \(x\)SbO\(_{1.5}\) pseudo-binary \((0.05 \leq x \leq 0.20)\).
In pseudo-binary, the glass density increases with the augmentation of SbO$_{1.5}$/TeO$_2$.

From the result (see Table 1), it can be seen that values of density increase with the addition of Sb$_2$O$_3$ is obviously due the difference of Sb and Te atoms weights.

Infrared spectroscopy

The infrared spectra transmission of glasses compositions are given in Fig. 4. A tellurite network basically consists of TeO$_4$ trigonal bipyramids (TBP) units and TeO$_3$ trigonal pyramid (TP) units, each of which has a lone pair of electrons, while the constitution of binary glasses depends on the second metal oxides. Suzuki [33] reported that TBP units were converted to TP ones on barium and sodium tellurite glasses. He proposed a mechanism (Eqs. 1 and 2) of the formation of the TP units:

$$3\text{TeO}_4^{2-} + \text{O}_2^{2-} \rightarrow \text{O}_2^{2-}\text{Te} - \text{O} + 2\text{TeO}_3^{2-}\text{O}^{2-}$$  \hspace{1cm} (1)

$$\text{O}_2^{2-}\text{Te} - \text{O} + 2\text{TeO}_3^{2-}\text{O}^{2-} + 2\text{O}^{2-} \rightarrow 3\text{TeO}_5^{2-}$$ \hspace{1cm} (2)

where O$_{1/2}$ represents bridging oxygen. These two reactions proceed as the content of a network modifying oxide increases until all the oxygen atoms of the (TBP) units become non-bridging (Eq. 3).

$$\text{TeO}_4^{2-} + \text{O}^{2-} \rightarrow \text{TeO}_5^{2-}$$ \hspace{1cm} (3)

The three oxygens in the TeO$_5^{2-}$ units are equivalent.

The TeO$_2$ glass ($x = 0$) infrared spectrum is rather similar to $x$TeO$_2$ data including the typical broadening observed in glasses. TeO$_2$ vitrification is thus characterized by a redistribution of infrared intensities due to spatial rearrangement of TeO$_4$E units involving a decrease of TeO$_4$E units symmetry which explains that the band at 625 cm$^{-1}$ becomes predominant [33]. As the Sb$_2$O$_3$ proportion increases (Fig. 4), the major band shifts from 625 cm$^{-1}$($x = 0$) to 667 cm$^{-1}$ ($x > 0.05$) which is attributed to TeO$_3$E trigonal pyramid. The presence of shoulder around 780 cm$^{-1}$ for all studied glass compositions is the signature of TeO$_4$E trigonal pyramid. For $x = 0.20$ we observed a band at 750 cm$^{-1}$ nearly which attributed to TeO$_3$E group. The absorption band nearly 500 cm$^{-1}$ which slightly increases in intensity with Sb$_2$O$_3$ content can be assigned to Te–O–Te and Te–O–Sb bridging bonds which would increase the network connectivity in agreement with th $T_g$ increase. On the other hand, from the reference spectra lithium tellurite glasses the infrared broad absorption bands at around 610 cm$^{-1}$ are attributed to group vibration of TeO$_6$ [21, 34]; Therefore in our preparation glasses, we do not observed this band so there is no oxidation of Te$^{+4}$ to Te$^{+6}$.

Raman spectra

In tellurite glasses, XANES, X-ray absorption and Raman spectroscopy previously showed that tellurium is surrounded by oxygen atoms and generally localized in three types of sites with different geometries. For the lowest amounts of additional oxides the dominant tellurium site
are \([\text{TeO}_4]\) trigonal bipyramids which are axially elongated and partly connected to each other sharing one oxygen atom. When increasing the amount of additional oxides they progressively convert into \([\text{TeO}_3]\) regular trigonal pyramids via \([\text{TeO}_3_{1+1}]\) entities where one axial \(\text{Te}–\text{O}_{\text{ax}}\) distance is elongating while the others shortens getting closer to the shortest equatorial \(\text{Te}–\text{O}_{\text{eq}}\) distances [25, 28–30, 35–41].

The Raman spectra of \(x\text{SbO}_{1.5}, (1–x)\text{TeO}_2\) \((5 \leq x \leq 20\%\,\text{mol})\) glasses are shown in Fig. 5a. For all samples, spectra obtained from different spots are identical showing high homogeneity of glasses. They are two pronounced peaks occur around 640–670 and 760–770 \(\text{cm}^{-1}\). The most prominent band at 659 \(\text{cm}^{-1}\) in the spectrum of pure glass is related to the combined vibrations of asymmetric stretching of \(\text{Te}_{\text{eq}}–\text{O}_{\text{ax}}–\text{Te}\) bonds and symmetric stretching of \(\text{TeO}_4\) (TBP). With addition of \(\text{SbO}_{1.5}\) up 20\% mol fraction, intensity of this band decreases (G1), while bands at 760–768 \(\text{cm}^{-1}\) (G2) attributed to stretching vibrations of non-bridging \(\text{Te}–\text{O}–\) bands in \(\text{TeO}_3\) (TP). The peak (G2), which is assigned to a stretching vibration of \(\text{TeO}_4\) units, was observed to decrease as the \(\text{Sb}_2\text{O}_3\) contents increases. The decrease in intensity would suggest the possibility of conversion from \(\text{TeO}_4\) TBP units to the other basic structural unit [37]. The peak (G1) is reported to be due to the perturbation of \(\text{TeO}_4\) (TBP) units into \(\text{TeO}_3\) (trigonal pyramids) units via the intermediate coordination of \(\text{TeO}_3_{1+1}\) [34, 35, 37]. Both features would clearly indicate that the network of the \(\text{TeO}_3\) structural unit increases with the increasing of \(\text{Sb}_2\text{O}_3\) contents. Other peaks around \((\text{P})\) 452–456 \(\text{cm}^{-1}\) are observed to be less sensitive to the \(\text{Sb}_2\text{O}_3\) contents. Antimony atoms are incorporation in the glass implied the formation of \(\text{Te}–\text{O}–\text{Sb}^\text{III}\) bridging bonds which stabilizes the glass formation in accordance with \(T_c\) – \(T_g\) increase. A decrease in the peak intensity would suggest the occurrence of the destruction of \(\text{Te}–\text{O}–\text{Te}^\text{IV}\) (or \(\text{O}–\text{Te}–\text{O}\)) in the linkages, thus resulted in the decreasing of the \(\text{Te}–\text{O}–\text{Te}\) linkages in a continue network of \(\text{TeO}_n\) \((n = 4, 3 + 1, \text{ or } 3)\) entities, which is consistent with the observation reported elsewhere [35], the intensity of this band decreases, while bands at 760 and 769 \(\text{cm}^{-1}\) were attributed to stretching vibrations of non-bridging \(\text{Te}–\text{O}–\) bands in \(\text{TeO}_3\) (TP) grow in intensity.

The spectral deconvolution of binary glasses indicates that five strong bands are present at about 450–767 \(\text{cm}^{-1}\). The bands are mainly attributed to the vibrations of coordination polyhedra of tellurium atoms. Figure 5b and Table 2 show results of band deconvolution of the spectra of \(x\text{SbO}_{1.5}, (1–x)\text{TeO}_2\) \((5 \leq x \leq 20\%\,\text{mol})\). A good agreement was obtained between the observed and simulated spectra. The fitting of the spectra was made with focus, a curve fitting soft wave especially adapted for analysis of optical spectra [42].

The orthotellurate ion, \(\text{TeO}_6^{6–}\), will have octahedral symmetry but may be strongly distorted. Vibrational modes for the tellurate anion should occur in the 620–650 \(\text{cm}^{-1}\) and in the 290–360 \(\text{cm}^{-1}\) regions [43]. In our spectrum, these intense bands do not appear, therefore there is no \(\text{Te}^6^{6–}\) in our vitreous composition.

**Crystalline phases**

A solid state investigation of the \(\text{Bi}_2\text{O}_3–\text{Sb}_2\text{O}_3–\text{TeO}_2\) system allowed synthesis of crystalline phases \(\text{SbTe}_3\text{O}_8\) and
Sb$_2$Te$_2$O$_9$ which have been obtained at 600–750 °C in air and characterized by XRD.

SbTe$_3$O$_8$

This phase, is characterized by powder diffraction X and indexed in the cubic system with parameter $a = 11.025(2)$ Å. No significant change in weight was observed, result implies no oxidation of Te$^{IV}$. This phase derived from fluorine phase is seems isotopic to TiTe$_3$O$_8$ [44] and its structure determination well be published.

Sb$_2$Te$_2$O$_9$

This phase is obtained from 2 mol TeO$_2$ and 1 mol Sb$_2$O$_3$. The mass gain during the synthesis of this compound was observed, which is probably related to an increase in oxygen content due to the oxidation of Te$^{IV}$ to Te$^{VI}$ and/or Sb$^{III}$ to Sb$^{V}$. Using the 11 most intense reflections of X-ray diffraction powder pattern, the indexing program dicvol [45] yielded monoclinic symmetry. All observed reflections were indexed and the figures merit were $M_20 = 38$. After a least-squares refinement, the cell parameters were:

\[
\begin{align*}
a & = 21.466(1) \text{ Å}, \\
b & = 4.903(1) \text{ Å}, \\
c & = 14.469(1) \text{ Å}, \\
\beta & = 110.89(1)\end{align*}
\]

These parameters were good agreement as reported in the ICDD files n°79-2317.

A series of compositions in the system of carefully chosen and were treated at different temperatures between 600 and 800 °C. Their analysis by X-ray diffraction revealed the existence of a stable phase BiSbO$_4$ [46] and other phases localized in Bi$_2$O$_3$–TeO$_2$–Sb$_2$O$_3$ (Sb$_2$O$_5$) pseudo-binary. Typical X-ray diffraction patterns of Bi$_{1-x}$Sb$_{1-x}$Te$_2$O$_4$ $0 \leq x \leq 0.1$ are shown in (Fig. 6). Solid solution of Bi$_{1-x}$Sb$_{1-x}$Te$_2$O$_4$ exist for the range (0 $\leq x \leq 0.1$) (compositions B and C) and the lattice parameters from XRD pattern are listed in Table 3. They are compared to BiSbO$_4$ phase. We find that the coupled substitution of antimony and bismuth atoms in size by the average of tellurium in the network and has no significant influence on

Table 2 Wave number of the Raman spectra for (1$-x$) TeO$_2$, $x$SbO$_{1.5}$ (0.05 $\leq x \leq 0.20$)

| Compositions | Wave number (cm$^{-1}$) | Intensity |
|--------------|------------------------|-----------|
| $x = 0.05$   | 456.778 (P) 614.958 658.375 (G1) 706.841 769.854 (G2) | 1.39923 0.822273 1.61736 0.656997 0.589822 |
| $x = 0.1$   | 454.85 (P) 615.501 658.321 (G1) 703.273 761.015 (G2) | 1.33616 0.834616 1.56564 0.477487 0.772023 |
| $x = 0.15$ | 455.516 (P) 618.24 658.542 (G1) 703.606 769.476 (G2) | 1.44533 0.865094 1.43989 0.692268 0.74740 |
| $x = 0.2$  | 456.836 (P) 614.482 658.103 (G1) 703.506 764.53 (G2) | 1.48301 0.788364 1.40338 0.547722 0.842251 |

Fig. 6 XRD patterns of Bi$_{1-x}$Sb$_{1-x}$Te$_2$O$_4$ solid solution with $0 \leq x \leq 0.1$

Table 3 Parameters evolution of Bi$_{1-x}$Sb$_{1-x}$Te$_2$O$_4$ solid solution with $0 \leq x \leq 0.1$

| Composition: Bi$_{1-x}$Sb$_{1-x}$Te$_2$O$_4$ | Cell parameters | Volume ($\pm 0.02$ Å$^3$) |
|-------------------------------------------|-----------------|--------------------------|
| $x = 0$: BiSbO$_4$ | $a = 5.469(1)$ Å, $b = 4.8847(1)$ Å, $c = 11.8285(1)$ Å, $\beta = 101.13^\circ(1)$ | 309.96 |
| $x = 0.05$: Bi$_{0.95}$Sb$_{0.05}$Te$_2$O$_4$ | $a = 5.469$ Å(1), $b = 4.8871(1)$ Å, $c = 11.822(1)$ Å, $\beta = 101.12^\circ(1)$ | 310.05 |
| $x = 0.10$: Bi$_{0.9}$Sb$_{0.1}$Te$_2$O$_4$ | $a = 5.466$ Å(1), $b = 4.884(1)$ Å, $c = 11.825(1)$ Å, $\beta = 101.19^\circ(1)$ | 309.75 |
the evolution of lattice parameters. Phase up to $x = 0.5$ (BiSbTeO$_4$) (A) adopt the Bi$_2$Te$_2$O$_7$ and the limit solid solution.

**Remarks**

Owing to the oxidation of Sb$^{+3}$ and Te$^{+4}$, the investigated Bi$_2$O$_3$–Sb$_2$O$_3$–TeO$_2$ system cannot be considered as a ternary, but rather as a pseudo-quaternary Bi$^{3+}$/Te$^{4+}$/Te$^{6+}$/Sb$^{+3}$/Sb$^{+5}$ system. Our investigation of the Bi$_2$O$_3$–Sb$_2$O$_3$–TeO$_2$ system revealed the important influence of the oxygen atmosphere on the chemistry and the crystallography of the phases found.

**Conclusions**

In the Bi$_2$O$_3$–Sb$_2$O$_3$–TeO$_2$ pseudo-ternary system, stable and transparent glasses were been synthesized at 800 °C. The vitreous domain in pseudo-binary (1–$x$)TeO$_2$–$x$Sb$_2$O$_3$ system is: 0.05 ≤ $x$ ≤ 0.2. Its characteristic temperatures (glass transition and crystallization temperatures) have been determined. The crystallization of the samples rich of Sb$_2$O$_3$ ($800–850$ °C) and the existence a solid solution with the formulation Bi$_{1–x}$Sb$_1x$Te$_2$ occurs for the investigated glasses. The formation Bi–O–Te linkages and 3-fold coordinated oxygen atoms which increase the polymerization of the glass network in accordance with an increase of the glass transition temperature which proportion depends on composition. A solid state investigation by X-ray diffraction in the ternary system allowed the existence a solid solution with the formulation Bi$_{1–x}$Sb$_1x$Te$_2$O$_4$ isotopic to BiSbO$_4$:0 ≤ $x$ ≤ 0.1.

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