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Influence of modifiers on thermal and optical properties of TeO_2–P_2O_5–ZnO–PbF_2 glasses

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A series of fluorotellurite glasses based on $70TeO_2$–$5M_xO_y$–$10P_2O_5$–10ZnO–$5PbF_2$ in mol%, where $M_xO_y = (WO_3, Nb_2O_5)$ doped with 2400 ppm of Er_2O_3 have been prepared by the conventional melt quenching method. The influence of modifiers on thermal and optical properties of glasses has been analyzed. Thermal characteristics of glasses like the glass transition temperature T_g, the temperature for the crystallization onset T_x, the maximum crystallization temperature T_c, and the thermal stability parameter were determined by the DSC method. The ellipsometric data have provided Sellmeier-type dispersion relations of the refractive index of the investigated glasses. The optical parameters are used to calculate the molar refractivity, molar polarizability, oxide ion polarizability, molar cation polarizability, and a number of polarizable atoms per unit volume for every glass composition in order to interpret the refractive index of these glasses.

Keywords: fluorotellurite, thermal stability, refractive index, luminescence, Judd–Ofelt theory.

1. Introduction

Due to a significant increment in the Er^{3+}-doped materials, the present paper aims to report newly developed fluorotellurite glasses doped with Er_2O_3 and to examine the effect of modifiers on the thermal and selected optical properties of the obtained glasses. Fluorotellurite glasses possess some advantages of both fluoride and tellurite

glasses, such as lower phonon energy, longer red-cutting edge, and excellent chemical stability as compared to other types of glasses [1, 2]. One can say that among all oxide glasses, the tellurite glasses have the poorest thermal stability due to their low value of glass transition temperature and therefore this kind of glasses is not suitable for use in mid-infrared photonics. On the other hand, their high rare earth solubility and high transparency in a wide wavelength range make the tellurite glasses very attractive from the point of view of possible applications [3–5].

However, one of the main drawbacks of tellurite glasses is the presence of hydroxyl (OH^-) groups that quenches radiative emission of rear earth (RE) ions [6]. Thus by addition of fluorine to the tellurite glass matrix it seems to be possible to reduce the OH^- concentration and the electron–phonon coupling.

In last years, extensive studies have been made to fabricate tellurite glass with low OH^- concentration. Most of this research is focused on the dehydration by incorporating fluoride raw materials [7–10].

There are many fluorides which were incorporated into the tellurite glass matrix, *e.g.* ZnF_2, NaF and BaF_2 [1, 11–13]. An interesting characteristic of the fluorotellurite glasses is the appearance of variations in their structural, thermal and optical properties when different modifier oxides (MgO, WO_3, CaO, BaO, SrO, CdO, and PbO) are introduced. In addition, TeO_2 together with other heavy metal oxides like Bi_2O_3, Sb_2O_3 or Nb_2O_5 produces glasses with some enhanced properties [14]. The more so, when Nb_2O_5 and ZnO are introduced into the tellurite glass network, thermal stability, chemical resistance and nonlinear optical performances are highly improved. Therefore, the vitreous system with composition containing TeO_2 together with Nb_2O_5 and ZnO has received great attention owing to the possibility of its use for several practical applications [15]. This paper examines the effect of selected matrix modifiers on thermal and optical properties of a new fluorotellurite glass prepared using the melt quenching method.

2. Experiment

In this work, the glasses from the system: $70TeO_2–5M_xO_y–10P_2O_5–10ZnO–5PbF_2$ in mol%, where M_xO_y = (WO_3, Nb_2O_5) are presented. The glasses have been doped with erbium ions. In order to introduce the Er^{3+} ions into the glass matrix, the respective oxides of an amount of 2400 ppm of Er_2O_3 have been added to the batches.

The following raw materials were used to prepare the batches: tellurium oxide (TeO_2), fluorite lead (PbF_2), zinc oxide (ZnO), tungsten trioxide (WO_3), niobium oxide (Nb_2O_5) and erbium oxide (Er_2O_3). Before melting, raw materials were pre-heated at 200, 300 and then 400°C, to eliminate or minimize the water content already present in the raw materials. The batches were put in a covered gold crucible and heated in a melting furnace to a temperature of 850°C for 30 min; the melt was stirred from time to time. The melt was poured out onto a graphite mold. Subsequently, the sample was transferred to an an-

T a b l e 1. The composition of investigated glasses.

	Sample M4	Sample M7	Sample M10
TeO_2 [mol%]	70	70	75
Nb_2O_5 [mol%]	5	–	–
WO_3 [mol%]	–	5	–
P_2O_5 [mol%]	10	10	10
ZnO [mol%]	10	10	10
PbF_2 [mol%]	5	5	5
Concentration of Er^{3+} [10^{19} cm^{-3}]	4.09	4.02	3.97

nealing furnace and kept for 2 h at 320°C, *i.e.* 15°C below the glass transition temperature T_g. Then the furnace was switched off and the glass sample was allowed to cool. The compositions of the investigated glasses are listed in Table 1.

The thermal stability of glasses obtained against crystallization was characterized in terms of the temperature interval $\Delta T_c = T_c - T_g$, where T_c is the onset of first exothermal crystallization event. The glass forming tendency parameter (Hruby parameter) $K_H = (T_c - T_g)/(T_m - T_c)$, with T_m, the melting temperature, was calculated. The characteristic temperatures of glass were determined by DSC measurements conducted on the NETZSCH 5 System operating in the heat flux DSC mode at a heating rate of 10°C/min. The density of glasses was measured according to the Archimedes principle using water as an immersion liquid. Concentration of Er^{3+} ions in the studied glass was calculated on the basis of density measurements.

For optical measurements, the annealed glass samples were sliced and polished to dimensions of about $10 \times 10 \times 2$ mm^3. The ellipsometric data were collected with a M-2000 Woollam ellipsometer in the spectral range 190–1700 nm. Knowledge of Ψ and Δ allows one to determine not only the dispersion of the optical constants, but also the roughness σ of a glass [16]. The samples have been measured for three angles of incidence, namely 60°, 65° and 70°. To analyze the data, we have combined all the angular spectra and we have fitted all the data simultaneously. The data have been analyzed using CompleteEASE 4.1 software.

3. Results and discussion

3.1. Thermal properties

In a glass of composition of $70TeO_2$–$5M_xO_y$–$10P_2O_5$–$10ZnO$–$5PbF_2$ in mol%, where M_xO_y = (WO_3, Nb_2O_5) doped with 2400 ppm Er_2O_3, presented in this study, tellurium oxide is a dominant component which plays the vital role as a conditional glass former. A chemical compound such as lead fluoride was added in order to tune specific properties such as minimization of OH^- content and improvement of thermal stability, respectively. ZnO is responsible for the depolymerization of the amorphous glass structure by increas-

ing TeO_3 units. By contribution of the network, former P_2O_5 several advantages are to be expected, *e.g.* low melting and softening temperatures and high UV transmission. Moreover, the presence of P_2O_5 makes glasses more suitable for RE doping.

The prepared fluorotellurite glasses have different colors ranging from yellowish to greenish depending on the kind of modifiers. All glass obtained were transparent and homogenous, and amorphous, according to XRD data. The prepared samples were air bubbles free and, based on the visual inspection, the defects were not observed. The results of the glass densities measurements are presented in Table 2. Measurements have been made for the variation of the densities of the two glass systems with different modifiers, *i.e.* 5 mol% of Nb_2O_5 and 5 mol% of WO_3 (glasses M4 and M7, respectively) at the expense of 5 mol% TeO_2 (glass M10). Their densities were found to vary in the range 5.13–5.34 g/cm^3. From Table 2 it is clear that the replacement of TeO_2 with WO_3 or Nb_2O_5 has a strong influence on the thermal stability, crystallization temperature T_x as well as T_g that shifts significantly to higher temperatures (Fig. 1).

The glass transition temperature is ranged from 343 to 366°C. It is noticed that the value of T_g increases by replacing TeO_2 with WO_3 and Nb_2O_5 modifiers, that may be due to the high strength of the bonds. The glass formation process is treated taking into account bound strength considerations. Usually in inorganic chemistry, Pauling's electronegativity and ionicity values are universally accepted as standardised characteristics of bonds and chemical interactions of atoms. We would like to explain the changes in the glass transition temperature values with use of similar factors charac-

T a b l e 2. Thermal characteristic of glasses (T_g – glass transition temperature, ΔC_p – the specific heat, T_c – the onset of crystallization temperature, and T_m – melting point).

Sample	T_g [°C]	ΔC_p [J g^{-1} K^{-1}]	T_c [°C]	ΔH_c [J g^{-1}]	$\Delta T_c = T_c - T_g$ [°C]	$K_H = \frac{T_c - T_g}{T_m - T_c}$	T_m [°C]	ρ [g/cm^3]
M10	343	0.164	433	84	90	0.45	631	5.2599
M7	357	0.183	484	34	127	1.10	599	5.3378
M4	366	0.159	504	32	138	1.40	602	5.4222

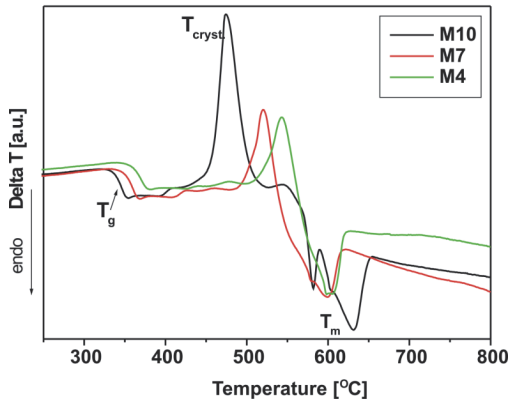

Fig. 1. DSC curves of glasses obtained.

terising crystallochemical properties of cations and atoms which have been introduced by GÖRLICH [16]. The effective nuclear charge of atomic core, defined as $Z_{\text{eff}} = \sqrt{I'_{\text{v}}}$, where I'_{v} is the ionisation energy in the Rydberg units, is needed to remove the valence electrons from an atom. Its values are closely related to Pauling's electronegativity. The relative difference of the nuclear charge of the two bonding atoms constitutes the factor determining the ionicity of the heteropolar bond. According to the Görlich scale [17], the ionicity i_{G} value of the bonds of the component atoms with oxygen is a parameter characterizing the strength of the bonds which increases with decreasing ionicity. The ionicity i_{G} or asymmetry of the chemical bonds between different atomic cores or ions, in a cation-oxygen bond, is measured by the relation $i_{\text{G}} = 1 - Z_{\text{eff1}}/Z_{\text{eff2}}$, where $Z_{\text{eff1}} < Z_{\text{eff2}}$. Intrinsic affinity between two different atoms forming a heteronuclear compound is directly connected with the value of ionicity of a chemical bond [18]. At the same time, the localization of the bonding electron $L = \overline{Z_{\text{eff}}} = (Z_{\text{eff1}} Z_{\text{eff2}})^{1/2}$ is assumed as a measure of the rigidity of the bonds. As $\overline{Z_{\text{eff}}}$ values describe the degree of localization of the bonding electrons, we may use for it the symbol L = localization. The value of L increases with the covalence of the bonds with oxygen [17, 18]. This parameter has been accepted as a measure of the rigidity of the bonds. The ionic character of W–O bonds ($i_{\text{G}} = 0.410$, $L = 2.447$), Nb–O bonds ($i_{\text{G}} = 0.395$, $L = 2.479$) replacing the covalent bonds such as Te–O bonds ($i_{\text{G}} = 0.285$, $L = 2.696$) made the glass structure less rigid, the consequence of which was a decreased stress in the glass. Its relaxation required more energy and hence higher T_{g} value with the substitution of modifiers (WO_3, Nb_2O_5) in the structure of glasses. The lower T_{g} value for M10 glass compared with that for M4 and M7 glasses indicates that the glass networks formed by these systems are stronger than those of M10 glass; this confirms the role of transition metal oxides Nb_2O_5 and WO_3 in forming the glass series as conditional glass network formers, as well as the role of the modifiers in forming a stable glass network [19]. The addition of WO_3 and Nb_2O_5 oxides to the glass structure produces complementary effects on magnitude of ΔC_{p}. As a result of structure depolimerization, the viscosity of the melt increases. Such a kind of behavior of glass melts might tend to the formation of more "ideal glass" in the sense of thermodynamics (lower entropy magnitude). But at the same time, the degraded units produce defects in which the oxygen is more tightly bonded to the central cation, thus the number of independent oscillators and the C_{p} value are increased. Based on DTA curves, it can be found that the onset crystallization temperature is shifted towards higher temperatures and its enthalpy ΔH_{n} becomes reduced. This is the evidence of the decreasing ability of the glass for crystallization, manifested by increased values of the index of thermal stability of the glass $\Delta T = (T_{\text{n}} - T_{\text{g}})$ [20, 21]. Moreover, the Hruby parameter K_{H}, calculated for M4 and M7 glasses, increases in comparison with K_{H} for M10 glass, confirming thus their higher stability against crystallization. The thermal stability values of glass M4 ($\Delta T = 138$) and glass M7 ($\Delta T = 128$) modified with Nb_2O_5 and WO_3 have similar values in comparison to the thermal stability values for the glass from the P_2O_5–ZnO–BaF_2–K_2TeO_3–Al_2O_3–Nb_2O_5–Er_2O_3 system presented in [22]. On the other hand, it should be noted that the same glass matrix $70TeO_2$–$5M_xO_y$–$10P_2O_5$–10ZnO–$5PbF_2$ in mol%, where M_xO_y = (SrO, BaO,

MgO) doped with 600 ppm Er_2O_3 reveals much lower thermal stability values (ΔT = 105, 106, 110) [23].

3.2. Optical properties

The aims of this part of the study were to assess the effect of the glass composition on the optical properties, namely the infrared (IR) transmission and refractive index of selected tellurite glasses. It is to be expected that the addition of WO_3 and Nb_2O_5 oxides to the TeO_2–P_2O_5–ZnO–PbF_2 glass structure can effectively enhance the optical properties. In Fig. 2 we present transmission and reflection spectra of all tested glasses.

The transmission for the niobium oxide sample is the largest in the range from 300 to 2500 nm. Addition of tungsten oxide results in the decrease of transmission com-

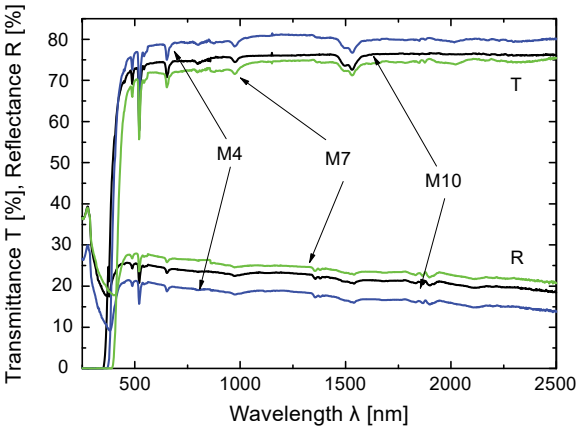

Fig. 2. Transmission T and reflection R spectra of tested glasses.

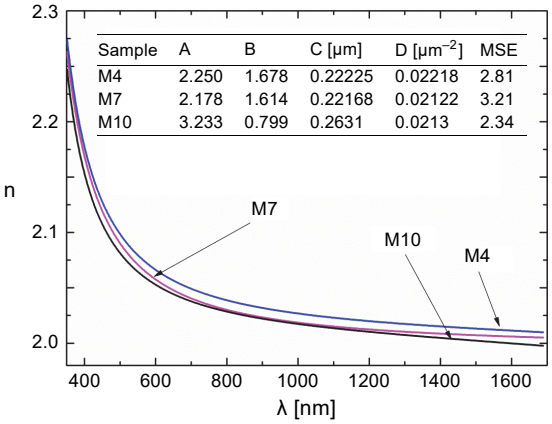

Fig. 3. Dispersion of the refractive index n of fluorotellurite glasses.

pared to the basic glass. Small peaks that are seen in the spectra are due to the presence of erbium ions in the glasses.

The ellipsometric measurements have allowed one to determine the dispersion of the refractive index n of the investigated glasses (Fig. 3).

There are many factors which have very important effects on the refractive index, such as density, polarizability of the first neighbor ions coordinated with it (anion), coordination number of the ion, electronic polarizability of the oxide ion, and optical basicity [24].

The refractive index of fluorotellurite glasses obtained is not lower than 2.0 in the range of 300–1700 nm. By incorporation of WO_3 or Nb_2O_5 oxides to the TeO_2–P_2O_5–ZnO–PbF_2 glass matrix, that greatly improves the thermal stability, the values of n slightly increased. Moreover, a linear correlation between n and density ρ is observed. The glass with composition $70TeO_2$–$5Nb_2O_5$–$10P_2O_5$–10ZnO–$5PbF_2$ (M4) has the highest value of the density ($\rho = 5.422$ g/cm^3) and the glass $70TeO_2$–$5WO_3$–$10P_2O_5$–10ZnO–$5PbF_2$ (M7) has a slightly lower value of the density (5.338 g/cm^3). Otherwise, $75TeO_2$–$10P_2O_5$–10ZnO–$5PbF_2$ (M10) has the lowest value of density ($\rho = 5.260$ g/cm^3). This change of density runs parallel to the change in the atomic mass of the modifier. The values of the refractive index n, density ρ, molar volume V_m, oxygen packing density (OPD), molar refraction R_m and the molar polarizability α_m are given in Table 3. The electronic polarizability of the glasses was evaluated using the following Lorentz–Lorenz equation giving the relationship between the molar refraction, the refractive index and density:

$$R_m = \left(\frac{n^2 - 1}{n^2 + 2}\right) V_m \tag{1}$$

T a b l e 3. Values of the refractive index n, density ρ, molar volume V_m, oxygen packing density (OPD), molar refraction R_m and the molar polarizability α_m.

Sample	Modifier type	ρ [g/cm^3]	V_m [cm^3]	V_o [cm^3/mol]	OPD	λ [nm]	n	α_m	α_m/V_m
M4	Nb_2O_5	5.4222	29.430	13.079	76.456	435.8	2.1392	6.344	0.216
						546.1	2.0804	6.136	0.208
						589.3	2.0686	6.093	0.207
						632.8	2.0596	6.059	0.206
M7	WO_3	5.3378	29.577	13.756	72.695	435.8	2.1309	6.347	0.215
						546.1	2.0719	6.135	0.207
						589.3	2.0599	6.091	0.206
						632.8	2.0509	6.057	0.205
M10	none	5.2599	29.328	13.965	71.607	435.8	2.1180	6.249	0.213
						546.1	2.0653	6.060	0.207
						589.3	2.0549	6.021	0.205
						632.8	2.0469	5.992	0.204

where V_m is the molar volume which is equal to

$$V_m = \frac{\sum x_i M_i}{\rho_{glass}} \tag{2}$$

where M_i is the molecular weight of the i-th component, x_i is the molar fraction of the i-th component and ρ_{glass} is the glass density.

Molar refraction R_m and refractive index n depend on polarizability α of the material by the sum of R_1 and R_m and are proportional to α as follows:

$$\alpha_m = \frac{3R_m}{4\pi N_A} \tag{3}$$

where α_m is the molar polarizability of glass and N_A is the Avogadro number.

The oxygen molar volume V_o has been calculated by the following expression:

$$V_o = \frac{\sum x_i M_i}{\rho_{glass}} \frac{1}{\sum x_i n_i} \tag{4}$$

where n_i is the number of oxygen atoms in each oxide.

From this data, it has to be noted that the value of R_m of the prepared glasses is in the range 18.393–19.319 cm^3 and α_m is in the range 6.249–6.347 at 435.8 nm. These values are slightly lower than the values of $75TeO_2$–$5WO_3$–$15Nb_2O_5$–$5M_xO_y$ glasses presented in [24] but, on the other hand, higher that those calculated for xRO–$(100 - x)TeO_2$ glasses [25]. Sample A with modifier CuO has the highest values of R_m and α_m, whereas sample H with modifier Na_2O has the lowest values of R_m and α_m.

In contradiction to the data presented in [24], the change in V_o values runs parallel to the change in the density value, but the value of V_m indeed is reduced along with a reduction in density, but the changes are not proportional. The changes in V_m and V_o values must be considered by taking into account the molecular weight of constituents in the glass composition, the number of oxygen atoms and bond length, and the cation radius and coordination number. The glass with addition of Nb_2O_5 has the highest values of V_m and V_o in comparison to the glass with WO_3 addition, due to the fact that Nb_2O_5 has higher molecular weight. The molar volume V_m values exhibits an increase with modifier addition at the expense of TeO_2 oxide. This result is due to the increase in the percentage of oxygen atoms which have the highest ionic radius in the glass structure. The oxygen packing density was calculated using the following expression [26]:

$$OPD = 1000\rho_{glass} \frac{\sum x_i n_i}{\sum x_i M_i} \tag{5}$$

As shown in Table 3, the oxygen molar volume and oxygen packing density values show opposite behavior to each other. Therefore, it can be concluded that, due to the

substitution of lower field intensity constituent ion Te^{4+} (1.40) with higher field intensity W^{6+} (1.47), a decrease in the oxygen molar volume and an increase in the oxygen packing density values are observed. This also results in high tightly packing of the glass network [27–29].

The molar polarizability of oxide ions is another factor affecting the refractive index value of the prepared glasses. The values of the molar polarizability obtained are shown in Table 3.

The relatively large refractive index of the tellurium niobate glasses is attributed to the hyper polarizability of Nb–O bonds [30]. The more polarizable the outer electrons, the higher n.

4. Conclusion

For all investigated glasses, the parameter $\Delta T = (T_n - T_g)$ is within the range from 90 to 137°C, indicating that the thermal stability against crystallization is very high. The addition of Nb_2O_5 and WO_3 is desirable to obtain a wide working temperature range for fiber drawing operation.

The refractive index of fluorotellurite glasses obtained is not lower than 2.0 in the range of 300–1700 nm. Addition of niobium oxide (Nb_2O_5) and tungsten oxide (WO_3) slightly raised the refractive index.

The transmission for the niobium oxide sample is the largest in the range from 300 to 2500 nm. Addition of tungsten oxide results in the decrease in transmission compared to the basic glass.

The glass with addition of Nb_2O_5 has the highest values of V_m (molar volume of the glasses) and V_o (oxygen molar volume) in comparison to the glass with WO_3 addition, due to the fact that Nb_2O_5 has higher molecular weight.

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