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Glass-forming region and optical properties of the TeO_2 – ZnO – NiO system

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ABSTRACT

The glass-forming region of the ternary TeO_2 – ZnO – NiO system has been determined using a conventional melt-quenching method under controlled condition. The stable samples containing up to 35 mol% NiO were prepared by the rapid cooling technique while a low cooling rate was allowed to obtain glasses containing 15 mol% NiO. The formation of tellurium, zinc and nickel complex oxides (e.g. $NiTe_2O_5$, $Zn_3Te_2O_8$) crystalline phases in a powder batches during the glass synthesis at different temperatures were studied. Investigations of the crystallization processes in glasses showed the formation of the same complex oxides phases as in the batch. The basic thermal properties of ternary glasses were determined by DSC and an increase of glass temperature with increase in the concentration of NiO in the glass matrix was shown. The optical absorbance spectrum of the glasses exhibited three broad bands with four maxima located at about 430, 720, 810 and 1320 nm corresponding to the d-d transitions of Ni^{2+} ion in octahedral coordination. The ligand field, splitting and Racah parameters were calculated and its dependence on the concentration of NiO was established.

1. Introduction

Glasses based on tellurium dioxide possess good thermal stability and chemical durability, high values of linear and nonlinear refractive index, a wide transmission window and an appropriate host material improving the luminescence of different rare earth ions [1–4]. These favorable properties make tellurite glasses as promising materials for use in nonlinear optical and photonic devices such as waveguide amplifiers, switches and storage devices [5].

Oxides of transition metals are known as network modifiers and they can change the structure of glasses affecting on thermal, magnetic and crystallization properties [6,7]. One of the most suitable compound for the aforementioned applications is nickel oxide [8]. It mostly exists in the divalent state [9] and is making them a perfect candidate for bandpass colored glass filters [10,11], the electrode in battery systems [12], also NiO has used also as a decolorizer in glasses containing high proportions of potassium oxide [13]. The octahedrally positioned Ni^{2+} ions in glasses can show three or four luminescence bands usually in the green, red and near-infrared regions [14], furthermore, they are capable of activating the luminescence of erbium ions [15,16] and laser emission are expected to exhibit with low threshold energy that is of great importance in telecommunication.

In recent years there has been much study focus on the research new tellurite glass systems possess high glass forming ability and improved optical and thermal properties. The binary zinc tellurite system is of great interest to glass technology because of the wide glass-forming region and the good solubility of transition oxide elements [17–21]. This allowed to prepare and investigate in detail multi components TeO_2 – ZnO – MO glasses, containing *s*- and *p*-elements [22,23]. As concerning *d*-elements in the tellurite glasses, their main physical, optical and thermal characteristics are limited to individual works [24–26]. The development of technology producing of optical materials requires suitable compositions for the manufacture of vitreous products. Concerning, the purpose of this paper was to establish the glass-forming area in the TeO_2 – ZnO – NiO system and study the physical, thermal and optical properties of the ternary glasses as a function of the nickel oxide mole fraction.

2. Experimental

2.1. Glass preparation

Tellurite glasses were prepared using the melt-quenching technique from high purity powders of TeO_2, ZnO and NiO. The oxides were weighed on a Shimadzu AUX320 electronic balance with an accuracy of 0.1 mg in suitable proportions, mixed and thoroughly ground in a porcelain mortar. Afterward, the 5 g batches were placed in a porcelain glazed crucible, melted in the air in a furnace at 800–1000 °C,

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Table 1
Glass transition (T_g), peak onset (T_x), crystallization (T_c) and melting temperatures (T_m) of the TeO_2 – ZnO – NiO glasses.

Sample ID	Glass compositions (mol%)			T_g, °C	T_x, °C	T_c, °C	T_m, °C	ΔT	K_H
	TeO_2	ZnO	NiO						
Te:Zn = 9:1									
TZN1	86.4	9.6	4.0	327	423	438	644	96	0.303
TZN2	82.8	9.2	8.0	340	418	453	637	78	0.263
Te:Zn = 4:1									
TZN3	76.8	19.2	4.0	333	454	477	644	121	0.389
TZN4	73.6	18.4	8.0	352	466	481	644	114	0.391
TZN5	70.4	17.6	12.0	375	453	473	637	78	0.298
Te:Zn = 7:3									
TZN6	67.2	28.8	4.0	346	455	470	628	109	0.387
TZN7	64.4	27.6	8.0	368	472	486	629	104	0.398
TZN8	61.6	26.4	12.0	390	492	505	–	102	–
Te:Zn = 3:2									
TZN9	57.6	38.4	4.0	367	475	488	–	108	–
TZN10	55.2	36.8	8.0	388	477	498	–	89	–
TZN11	52.8	35.2	12.0	406	503	525	–	97	–

depending on the percentage of NiO, and liquid was kept at this temperature for 15 min to ensure homogeneity. The melt was then cooled using two quenching rates. The first route, the melt was cast between two steel plates of high-alloy steel heated to 280 °C, this cooling rate was about 100 K/s. The prepared glass samples were annealed at 330 °C for 1 h to relieve thermal stress and then the furnace was switched off and the samples slowly cooled to ambient temperature. In the second route, glasses are performed by pressing melt between two polished heavy metal plates with an average cooling rate of about 10^3 K/s. The glassy materials obtained by these methods were polished to investigate the optical properties. The glass studied compositions and their specifications are listed in Table 1.

2.2. X-ray diffraction analysis

The amorphous and crystalline nature of samples was checked by X-ray diffraction (XRD) analysis with powdered samples on Shimadzu XRD-6000 diffractometer at the range of 2θ from 10 to 60° utilizing Cu K_α radiation with an applied voltage of the tube of 40 kV and 30 mA anode current at the rate of 2° min.

2.3. Differential scanning calorimetry (DSC)

Thermal properties of the synthesized glass were investigated by differential scanning calorimetry using the simultaneous system Netzsch STA 409 PC Luxx under flowing argon gas with a programmed heating rate of 10 °C per minute in the temperature range 30–700 °C in a platinum crucible. The glass transition temperature (T_g) was obtained by drawing tangents to the endothermic peak and the onset of crystallization (T_x) was taken as the temperature at which the heat flow starts to increase.

2.4. Optical properties

The optical absorption spectra of the glass samples were recorded at room temperature in the visible (VIS) and near-infrared (IR) regions using a double beam Shimadzu UV-3600 spectrophotometer in the wavelength range 310–2800 nm with a scanning step of 2 nm and a slit width of 8 nm. The transmission cut-off wavelength was determined by the linear extrapolation of the steep portion of the transmission curve to the intersection with the x-axis [27].

2.5. Physical properties of glasses

The density of glasses (ρ) was determined at room temperature by Archimedes principle using distilled water as immersion liquid (error for the determined was < 0.01 g/cm^3) and a digital balance of sensitivity 10^{-4} g. The density was obtained from the relation

$$\rho = \rho_b \frac{W_{air}}{W_{air} - W_{water}}, \quad (1)$$

where ρ_b is the density of distilled water, W_{air} и W_{water} – weight of the glass sample in air and in distilled water, respectively.

Theoretical density (ρ_{cal}) was calculated as the additive contribution of each oxide to glass by formula [28]

$$\rho_{cal} = \sum \rho_i x_i, \quad (2)$$

where ρ_i – density of each component i, x_i – the molar fraction of each component i.

The molar volume of glasses (V_m) was calculated as a function of the mole fraction of each component using the relation

$$V_m = \sum \frac{x_i M_i}{\rho}, \quad (3)$$

where x_i and M_i – the molar fraction and molecular weight of i-th component, ρ is density of the glass.

Oxygen molar volume (V_O) was calculated by the following formula:

$$V_O = V_m \left(\frac{1}{\sum x_i n_i} \right), \quad (4)$$

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

Oxygen packing density (OPD) was calculated by formula

$$\mathrm{OPD} = 1000C(\rho/M), \quad (5)$$

where C is the number of oxygen atoms per each composition, ρ is the density and M is the molecular weight of the glass sample.

2.6. Structural investigations

Average cross-link density ($\bar{n}_c$) of the glasses was calculated using the relation [28]:

$$\bar{n}_c = \frac{\sum x_i (n_c)_i (N_c)_i}{\sum x_i (N_c)_i}, \quad (6)$$

where x_i is the molar fraction of each component i; N_c is the number of

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