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## Author's Accepted Manuscript

Glass formation in the  $\text{As}_2\text{Se}_3\text{-As}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$  system

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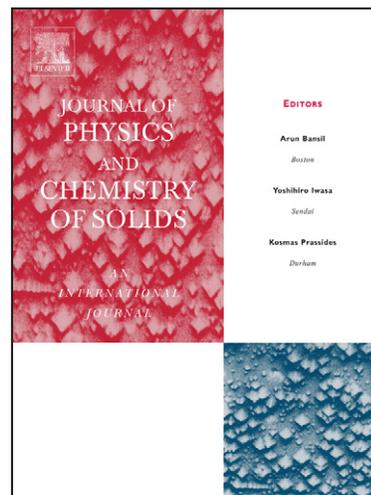
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**Glass formation in the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system****T. Hristova-Vasileva<sup>1</sup>, V. Vassilev<sup>1</sup>, L. Aljihmani<sup>1</sup>, S. Boycheva<sup>2</sup>**

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**Abstract**

Chalcogenide glasses from the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system were synthesized for the first time. The glass-forming region was determined by X-ray diffraction and electron microscopic analyses.

The basic physicochemical parameters such as density ( $d$ ), microhardness (HV) and temperatures of phase transformations (glass transition  $T_g$ , crystallization  $T_{cr}$  and melting  $T_m$ ) were measured. Compactness and some thermomechanical characteristics such as volume ( $V_h$ ) and formation energy ( $E_h$ ) of micro-voids in the glassy network as well as the elasticity module ( $E$ ) were calculated. The glass-forming ability was evaluated according to Hruby's criteria ( $K_G$ ). The correlation between composition and properties of the  $(\text{As}_2\text{Se}_3)_x(\text{As}_2\text{Te}_3)_y(\text{Sb}_2\text{Te}_3)_z$  glasses was established and comprehensively discussed.

Keywords: A. chalcogenides, B. synthesis, C. X-ray diffraction, D. thermodynamic properties

**Introduction**

The chalcogenide glasses can be obtained by joined melting of elements from the chalcogenides group (S, Se and Te) with other elements, most often from the V<sup>th</sup> (As, Sb) and 4<sup>th</sup> (Ge, Si) group of the Periodic table of elements. These glasses are unique due to their property to transmit light in the near and middle infra red parts of the spectra (from ~ 1 to > 18  $\mu\text{m}$  [1]) and by to this reason they are convenient for construction of IR-sensors, CO<sub>2</sub> lasers with application in the microsurgery, as well as for optical transport and storage media. These glasses are semiconductors with band gap values from 1 to 3 eV [1,2].

The As and the Te form glasses in a narrow concentration area, which is in the range from 42 to 52 at. % Te (when cooled on air) and 54 at. % Te (when quenched in cold water) [3].

The addition of third component to the As-Te system leads to enlargement of the glass forming region [4-12].

The glasses from the As-Te-Sb system are perspective material for the integral microelectronics, but the glass forming region in this system is very narrow [12], since there is no element in this system, which is a typical glass formator.

The introduction of Se will improve the glass formation in the As-Te-Sb system by widening the glass forming region and at the same time will not decrease significantly the mean molecule mass of the system. On the other hand the introduction of As and Sb in the form of their stable tellurides As<sub>2</sub>Te<sub>3</sub> and Sb<sub>2</sub>Te<sub>3</sub>, respectively, and of Se – in the form of As<sub>2</sub>Se<sub>3</sub> will keep this tendency. The transformation of the As-Te-Sb system, after substitution of a part of the tellurium with Se, into the As<sub>2</sub>Se<sub>3</sub>-As<sub>2</sub>Te<sub>3</sub>-Sb<sub>2</sub>Te<sub>3</sub> system presumes wider glass forming region (higher solubility of components

with near chemical and structural composition), higher crystallization stability, keeping to a certain degree the properties of the glasses from the elemental system, etc.

The aim of the present work is the outlining of the glass forming region of the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system and the determination of the basic characteristics of the glasses in it.

### Experimental procedures

The glass forming region in the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system was outlined by the help of 27 compositions – Table 1.

Table 1. Compositions used for outlining the glass forming region in the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system ( $m=y/(x+y)$ )

The starting compounds  $\text{Me}_n\text{Ch}_m$  (Me=As and Sb; Ch=Se and Te) and the glasses (4g) from the  $(\text{As}_2\text{Se}_3)_x(\text{As}_2\text{Te}_3)_y(\text{Sb}_2\text{Te}_3)_z$  system were synthesized by direct mono-temperature synthesis in sealed under residual pressure of  $1 \cdot 10^{-3}$  Pa quartz ampoules. The initial materials used for the synthesis of the  $\text{Me}_n\text{Ch}_m$  compounds were Se and Te with purity 5N (Fluka) and As and Sb – 3N5 (Merck). The synthesis characteristics (temperatures and duration of the isothermal steps, as well as heating rate between them) were in conformity to the physical and chemical features of the starting components, the intermediate and the final phases. At the maximum synthesis temperature of the glasses ( $700 \pm 10$  °C) a vibration stirring of the smelter with duration 2 hours was performed. The last was tempered at temperature of  $650 \pm 10$  °C and quenched in ice+water mixture with cooling rate  $10$ - $15$  °C  $\text{s}^{-1}$ .

The obtained bulk samples were investigated through XRD (diffractometer TUR-M16 with  $\text{CuK}\alpha$ -radiation and Ni-filter) and EMA (electronic microscope TEM Philips-3003).

The characteristic temperatures of the glasses were defined by differential thermal analysis (DTA) using apparatus from the F.Paulic-J.Paulic-L.Erdey system of the company MOM-Hungary at heating rate of  $10\text{ }^\circ\text{C min}^{-1}$ , standard substance –  $\alpha$ - $\text{Al}_2\text{O}_3$  and accuracy of  $\pm 5\text{ }^\circ\text{C}$ .

The density ( $d$ ) of the samples was measured with accuracy  $\pm 5\%$  at air temperature of  $25\text{--}30\text{ }^\circ\text{C}$  by the hydrostatic method using toluene as immersion fluid. The density of toluene is defined from the equation:  $d = 0.879 - 0.001T$ , where  $T$  is the temperature [ $^\circ\text{C}$ ], at which the measurement was made.

The microhardness (HV) was measured by the Vickers' method (a metallographic microscope MIM-7 with built in microhardnessmeter PMT-3 has been used at load of 10 g). The working values of HV are an average value of 20 measurements per sample; accuracy –  $\pm 5\%$ .

The elasticity module ( $E$ ), the minimal volume of the micro-voids ( $V_h$ ), the energy for their formation ( $E_h$ ) and the Hruby's criteria ( $K_G$ ) [13] were calculated by the Eqs. (1) and (2) [14]:

$$E = 15\text{HV}, V_h = 5.04 \frac{T_g}{\text{HV}}, E_h = 30.729T_g \quad (1)$$

$$K_G = \frac{T_{cr} - T_g}{T_m - T_{cr}} \quad (2)$$

where  $T_g$ ,  $T_{cr}$  and  $T_m$  are the temperatures of glass transition, crystallization and melting.

## Results and discussion

The observations with non-armed eye and binocular magnifier ( $4\times$ ) show that the synthesized samples from the  $\text{As}_2\text{Se}_3\text{-As}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$  system were dark colored, with strong luster and shell-like surface.

The results from the XRD show that some of the compositions (group a) are amorphous, without peaks on the diffractograms and with the characteristic X-ray amorphous plateau – these compositions form the glass forming region (Fig. 1). Other compositions (group b, Fig. 1b) show diffraction peaks with small intensity (compositions on the region's boundary). The diffractograms of the samples from group c show well expressed peaks. These samples are crystalline and get outside the region of glass formation (Fig. 1c).

The EMA investigations show smooth and homogenous surface for the samples from group a and for the compositions of group b small crystalline objects (micro-crystals) appear. The surface of the samples from group c is rough, without the characteristic for the glassy phase regions.

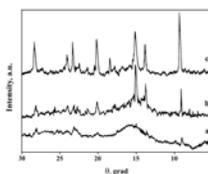


Fig. 1. Typical diffractograms of samples from the  $\text{As}_2\text{Se}_3\text{-As}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$  system: a) composition  $(\text{As}_2\text{Se}_3)_{63}(\text{As}_2\text{Te}_3)_{27}(\text{Sb}_2\text{Te}_3)_{10}$  (in the glass forming region); b) composition  $(\text{As}_2\text{Se}_3)_{32.8}(\text{As}_2\text{Te}_3)_{49.2}(\text{Sb}_2\text{Te}_3)_{18}$  (on the glass forming boundary); c) composition  $(\text{As}_2\text{Se}_3)_{55.8}(\text{As}_2\text{Te}_3)_{6.2}(\text{Sb}_2\text{Te}_3)_{38}$  (outside the glass forming region).

The region of glass formation in the  $\text{As}_2\text{Se}_3\text{-As}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$  system (Fig. 2), outlined on the basis of the results from the XRD and EMA analyses, is drawn to the  $\text{As}_2\text{Se}_3$ -rich region, lies fully on the  $\text{As}_2\text{Se}_3\text{-As}_2\text{Te}_3$  side (from 0 to 100 mol %  $\text{As}_2\text{Se}_3$ ) and partially on the  $\text{As}_2\text{Se}_3\text{-Sb}_2\text{Te}_3$  side (from 0 to 38 mol %  $\text{Sb}_2\text{Te}_3$ ). Glasses have not been obtained in the  $\text{As}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$  system.

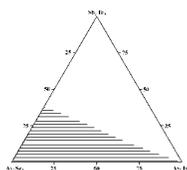


Fig. 2. Glass forming region in the  $\text{As}_2\text{Se}_3\text{-As}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$  system.

The investigated thermal characteristics  $T_g$ ,  $T_{cr}$  and  $T_m$  of the samples, defined by DTA, are shown in Table 2. Some samples typical derivatograms are presented at figure 3. The different effects could be seen on the thermograms. The first is connected to the glass transition temperature (change of the angle coefficient of the  $T(\tau)$  dependence); the second is exothermal with well expressed maximum and is related to the crystallization process; the last effect is endothermal and is obtained as result of the melting of already crystallized phase or solid solution.

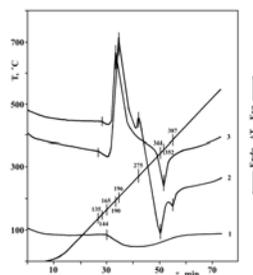


Fig. 3. Typical thermograms of samples from the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system: 1) sample with composition  $(\text{As}_2\text{Se}_3)_{81}(\text{As}_2\text{Te}_3)_9(\text{Sb}_2\text{Te}_3)_{10}$ ; 2) sample with composition  $(\text{As}_2\text{Se}_3)_{45}(\text{As}_2\text{Te}_3)_{45}(\text{Sb}_2\text{Te}_3)_{10}$ ; 3) sample with composition  $(\text{As}_2\text{Se}_3)_{63}(\text{As}_2\text{Te}_3)_7(\text{Sb}_2\text{Te}_3)_{30}$ .

The temperature of glass transition  $T_g$  varies in the range 125-165 °C (Table 2). It decreases slightly with the increment of the  $\text{As}_2\text{Te}_3$  content and relatively more strongly with the addition of  $\text{Sb}_2\text{Te}_3$ . This course of the  $T_g(m)$  and  $T_g(z)$  dependencies is logical, since the glass forming ability decreases in the row  $\text{As}_2\text{S}_3 \rightarrow \text{As}_2\text{Se}_3 \rightarrow \text{As}_2\text{Te}_3$ , while the  $\text{Sb}_2\text{Te}_3$  is a crystalline substance and has never been obtained in glassy state.

Table 2. Thermal properties and Hruby's criteria of glassy phases from the  $(\text{As}_2\text{Se}_3)_x(\text{As}_2\text{Te}_3)_y(\text{Sb}_2\text{Te}_3)_z$  system.

In dependence of the composition, the crystallization temperature  $T_{cr}$  varies in the range 177-229 °C (Table 2). On the thermogram of glass with composition  $(\text{As}_2\text{Se}_3)_{81}(\text{As}_2\text{Te}_3)_9(\text{Sb}_2\text{Te}_3)_{10}$  were not registered crystallization and melting effects which shows that this glass is stable and most probably  $\text{As}_2\text{Te}_3$  and  $\text{Sb}_2\text{Te}_3$  are built in the structure of the glass formator  $\text{As}_2\text{Se}_3$  in a way, which prevents the structural reorganizations leading to the amorphous-crystalline state transition. The lack of crystalline phase expels the existence of clearly presented endothermal melting effect. The stability of the glass determines the presence of "diluted" protuberance in a direction corresponding to the endoeffect.

For the compositions, corresponding to points 27, 7 and 12 (Table 1), situated near the glass formation border, two crystallization and melting temperatures were registered – Table 2. The presence of two exoeffects, corresponding to  $T_{cr}$ , is logical since the probability of crystallization of more than one phase (solid solutions based on  $As_2Te_3$  and  $Sb_2Te_3$ ) is fairly larger.

$T_{cr}$  depends slightly of the  $As_2Te_3$  concentration, since the  $As_2Se_3$  and  $As_2Te_3$  are isomorphous, and much strongly from the concentration of  $Sb_2Te_3$ , as the addition of this component to  $As_2Se_3$  requires more complicated changes in the structure of the glass: partial substitution of As- with Sb- and of Se- with Te-atoms.

The melting temperature  $T_m$  is in the range 320-377 °C (Table 2). The above-said for  $T_{cr}$  is valid also for the  $T_m$ , since  $T_m(Sb_2Te_3) > T_m(As_2Se_3) \cong T_m(As_2Te_3)$

The Hruby's criteria  $K_G$  gives qualitative idea for the glass forming ability of the alloys.  $K_G$  changes in narrow limits (Table 2), as with the increment of the content of  $As_2Te_3$  ( $z=const$ ) and  $Sb_2Te_3$  ( $m=const$ ) slightly decreases, which as it has been mentioned above, is due to the characteristics of the  $As_2Te_3$  and  $Sb_2Te_3$ .

The microhardness is in the range of 40-85 kgf/mm<sup>2</sup> and logically decreases with the increase of the  $Sb_2Te_3$  content, since  $HV_{Sb_2Te_3} \ll HV_{As_2Se_3} \cong HV_{As_2Te_3}$ , respectively 18 [15], 150 [2] and 160 kgf/mm<sup>2</sup> [16] - Table 3. Due to the same reasons the HV depends slightly of the  $As_2Te_3$  concentration.

Table 3. Physical and thermomechanical properties of glassy samples from the  $(As_2Se_3)_x(As_2Te_3)_y(Sb_2Te_3)_z$  system.

The density of the glasses varies in the range 4.80-5.35 g/cm<sup>3</sup> and logically increases with the increase of the As<sub>2</sub>Te<sub>3</sub> and Sb<sub>2</sub>Te<sub>3</sub> content ( $d_{As_2Se_3} = 4.75$  g/cm<sup>3</sup> [17];  $d_{As_2Te_3} = 6.5$  g/cm<sup>3</sup> [17];  $d_{Sb_2Te_3} = 6.57$  g/cm<sup>3</sup> [18]) - Table 3.

The compactness of the investigated glasses (Table 3) depends weakly of the Sb<sub>2</sub>Te<sub>3</sub> content (expressed by z at m=const), as well as of the As<sub>2</sub>Te<sub>3</sub>/As<sub>2</sub>Se<sub>3</sub> proportion (expressed by m=y/(x+y) at z=const). From the analysis of the compactness equation [14]

$$C = d \left\{ \sum_{i=1}^n \frac{M_i x_i}{d_i} - \sum_{i=1}^n \frac{M_i x_i}{d} \right\} \left[ \sum_{i=1}^n M_i x_i \right]^{-1} = d \sum_{i=1}^n \frac{M_i x_i}{d_i} \left( \sum_{i=1}^n M_i x_i \right)^{-1} - 1 = d.F_1 - 1$$

and rendering an account to the fact that  $M_{As_2Se_3} < M_{As_2Te_3} < M_{Sb_2Te_3}$  and  $d_{As_2Se_3} < d_{As_2Te_3} \cong d_{Sb_2Te_3}$ , a conclusion can be made, that the function  $F_1$  is decreasing. From this follows that if  $d.F_1 \cong \text{const}$ , than  $C_{m=\text{const}}(z)$  depends slightly from the composition and  $C_{z=\text{const}}(m)$  practically does not depend.

The elasticity modulus follows the path of HV and changes within the limits 594-1270 kgf/mm<sup>2</sup> - Table 3.

The volumes of the micro-voids is in the range  $8.0 \times 10^{-3}$ - $17.7 \times 10^{-3}$  Å<sup>3</sup> (Table 3).  $V_h$  decreases when increasing the As<sub>2</sub>Se<sub>3</sub> content (z=const) and increases with the increase of the Sb<sub>2</sub>Te<sub>3</sub> content (m=const). This way of  $V_h$  change from the composition is expected, since the exchange of As<sub>2</sub>Se<sub>3</sub> with As<sub>2</sub>Te<sub>3</sub> is connected to an exchange of Se<sup>-</sup> with Te<sup>-</sup> atoms, which ionic radii are  $r_{Se^{2-}} < r_{Te^{2-}}$  (1.91 Å < 2.11 Å). The alternation of  $V_h$  when switching the As<sub>2</sub>Se<sub>3</sub> (As<sub>2</sub>Te<sub>3</sub>) with Sb<sub>2</sub>Te<sub>3</sub>, since  $r_{As^{3+}} < r_{Sb^{3+}}$  (0.69 Å < 0.90 Å).

The energy for formation of micro-voids follows the path of  $T_g$  and varies from 3840 to 5000 kJ mol<sup>-1</sup> (Table 3).

### **Conclusions**

New chalcogenide glasses were synthesized and their glass forming region was outlined.

A number of thermal (temperatures of glass transition, crystallization and melting, Hruby's criteria), physicochemical (density, microhardness, compactness) and thermomechanical (elasticity module, micro-voids volume and energy for their formation) properties of the glassy phases were studied. A correlation between these properties and the composition of the glasses from the investigated system was established and a method for its explanation has been proposed.

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## Tables

Table 1. Compositions used for outlining the glass forming region in the  $\text{As}_2\text{Se}_3$ - $\text{As}_2\text{Te}_3$ - $\text{Sb}_2\text{Te}_3$  system ( $m=y/(x+y)$ )

No	Composition, mol %			m	State
	$\text{As}_2\text{Se}_3$	$\text{As}_2\text{Te}_3$	$\text{Sb}_2\text{Te}_3$		
1	9.5	85.5	5	0.9	Glassy + crystalline
2	18	72	10	0.8	Glassy + crystalline
3	25.5	59.5	15	0.7	Glassy + crystalline
4	32	48	20	0.6	Crystalline
5	37.5	37.5	25	0.5	Glassy + crystalline
6	45	30	25	0.4	Glassy
7	49	21	30	0.3	Glassy
8	52	13	35	0.2	Crystalline
9	58.5	6.5	35	0.1	Glassy
10	72	8	20	0.1	Glassy
11	56	24	20	0.3	Glassy
12	40	40	20	0.5	Glassy
13	81	9	10	0.1	Glassy
14	63	27	10	0.3	Glassy
15	36	54	10	0.6	Glassy
16	45	45	10	0.5	Glassy
17	32.8	49.2	18	0.6	Glassy + crystalline
18	26.1	60.9	13	0.7	Glassy
19	22.5	67.5	10	0.75	Glassy + crystalline
20	19	76	5	0.8	Glassy
21	9.7	87.3	3	0.9	Glassy
22	4.85	92.15	3	0.95	Crystalline
23	43.2	28.8	28	0.4	Crystalline
24	46.9	20.1	33	0.3	Crystalline
25	53.6	13.4	33	0.2	Glassy
26	55.8	6.2	38	0.1	Crystalline
27	63	7	30	0.1	Glassy

Table 2. Thermal properties and Hruby's criteria of glassy phases from the  $(As_2Se_3)_x(As_2Te_3)_y(Sb_2Te_3)_z$  system.

№	Composition			m	$T_g$	$T_{cr}$	$T_m$	$\Delta T$	$K_G$
	x	y	z						
13	81	9	10	0,1	165	-	-	-	-
10	72	8	20	0,1	149	209	361	60	0,3947
27	63	7	30	0,1	139	190/275	344/387	51	0,3566
14	63	27	10	0,3	163	210	330	47	0,3916
11	56	24	20	0,3	145	197	356	52	0,3270
7	49	21	30	0,3	131	178/270	349/425	47	0,2748
16	45	45	10	0,5	144	196	352	52	0,3333
12	40	40	20	0,5	125	177/262	377/454	52	0,2600
15	36	54	10	0,6	136	185	342	49	0,3121

Table 3. Physical and thermomechanical properties of glassy samples from the

 $(As_2Se_3)_x(As_2Te_3)_y(Sb_2Te_3)_z$  system.

№	Composition			m	$T_g$ , °C	HV, kgf mm <sup>-2</sup>	d, g cm <sup>-3</sup>	E, kgf mm <sup>-2</sup>	$V_h$ , 10 <sup>-3</sup> Å <sup>3</sup>	$E_h$ , kJ mol <sup>-1</sup>	C
	x	y	z								
13	81	9	10	0.1	165	76	4.56	1136	11.0	5070	-0.108
10	72	8	20	0.1	149	46	4.79	696	16.2	4579	-0.096
27	63	7	30	0.1	139	40	4.92	594	17.7	4271	-0.105
14	63	27	10	0.3	163	80	4.78	1194	10.3	5009	-0.120
11	56	24	20	0.3	145	47	5.06	698	15.7	4456	-0.092
7	49	21	30	0.3	131	40	5.21	600	16.5	4025	-0.872
16	45	45	10	0.5	144	82	5.12	1226	8.9	4425	-0.107
12	40	40	20	0.5	125	47	5.35	704	13.4	3841	-0.084
15	36	54	10	0.6	136	85	5.23	1271	8.1	4179	-0.113

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