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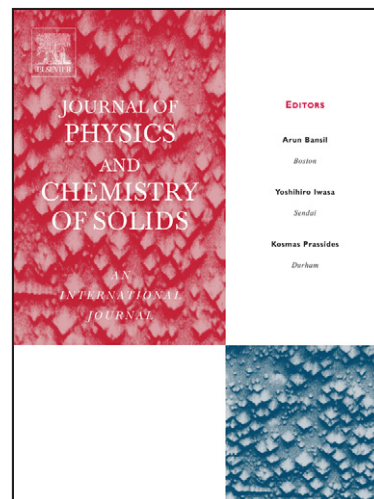
PII: S0022-3697(08)00025-5
DOI: doi:10.1016/j.jpcs.2008.01.010
Reference: PCS 5364

To appear in: *Journal of Physics and Chemistry of Solids*

Received date: 8 March 2007
Revised date: 4 November 2007
Accepted date: 11 January 2008

Cite this article as: V. Vassilev, I. Karadashka and S. Parvanov, New chalcogenide glasses in the $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3-\text{CdTe}$ system, *Journal of Physics and Chemistry of Solids* (2008), doi:10.1016/j.jpcs.2008.01.010

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NEW CHALCOGENIDE GLASSES IN THE $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3-\text{CdTe}$ SYSTEMV. Vassilev¹, I. Karadashka¹, S. Parvanov²

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Abstract

Chalcogenide glasses from the $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3-\text{CdTe}$ system were synthesized.

The basic physicochemical parameters such as density (d), microhardness (HV) and the temperatures of phase transformations (the 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 module of elasticity E were calculated. The overall mean bond energy $\langle E \rangle$, the mean coordination number Z , the mean bond energy of the average cross-linking/atom and the average bond energy per atom of the “remaining matrix” - \bar{E}_c and \bar{E}_{rm} , as well as the average heteropolar bond energy E_{hb} , the degree of „cross-linking/atom“ P_p , the radial bond strength were determined.

The correlation between the composition and properties of the $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3-\text{CdTe}$ glasses was established and comprehensively discussed.

Keywords: A. Chalcogenide glasses; A. Amorphous materials; B. Chemical synthesis

1. Introduction

Ion-selective electrodes (ISE) with membranes of chalcogenide glasses (CG) are becoming more widely used in an analytical practice. The membranes for heavy metals ion detection are produced by melting or by the pressing of insoluble metal-chalcogenides [1]. In comparison to polycrystalline chalcogenides, the glassy chalcogenides show more stability in respect to electrolyte dissolving and resistance in aggressive environments [2]. Because of these properties, CG are more often used as membrane materials for construction of ISE for Ag(I)-, Pb(II)-, Fe(III)-, Cu(II) detection, instead of metal-chalcogenides [2 ÷ 5].

In this respect, the development of new chalcogenide glasses on the basis of As_2Se_3 with metal-chalcogenides MeCh (Me=Zn, Cd, Hg, Co, Ni, Sn, Tl, Cr and others; Ch=S, Se and Te) with the aim of inventing new membrane materials is a prospective and interesting task. The research involves: 1) syntheses of new chalcogenide glasses; 2) investigation of their physicochemical properties, including density, microhardness, temperatures of glass transition, crystallization and complete melting, direct current (d.c) and alternating current (a.c) conductivity and others; 3) investigation of morphology using X-ray diffraction and mass-spectroscopy; 4) investigation of the composition using Auger electron spectroscopy [6].

The aim of the present report is to determine the glass-forming region and investigation of some, mainly physical-chemical characteristics, of chalcogenide glasses in the system $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3-\text{CdTe}$.

2. Experimental procedures

The glass-forming region was determined with the help of 20 synthesized compositions of $(\text{Ag}_2\text{Te})_x(\text{As}_2\text{Se}_3)_y(\text{CdTe})_z$, where x, y, z are the mol % ($x+y+z=100$) of Ag_2Te , As_2Se_3 and CdTe respectively - Table 1. The binary As_2Se_3 - CdTe system has been studied in our previously work [7]. Starting components were As, Te and Se with 5N purity and Ag and Cd with 4N purity.

The samples of the system Ag_2Te - As_2Se_3 - CdTe as well as the starting components Ag_2Te , As_2Se_3 and CdTe were obtained through a direct mono-temperature synthesis in vacuumed quartz ampoules under the following conditions: three-stage increase of temperature to 650, 850 and 1100 °C with velocity 3-4, 2-3 and 2-3 °C min^{-1} and homogenized annealing at these temperatures 0.5, 1 and 2 h respectively combined with continuous vibrational stirring of the melt at the last-mentioned temperature. Followed by a reduction of the temperature to 900 ± 10 °C and applying continuous vibrational stirring of the melt as it was annealed for 0.5 h and after that the melt was cooled in a mixture of water and ice.

The bulk glasses were analyzed by a visual, X-ray phase analysis (diffractometer TUR-M62 with CuK_α radiation and a Ni filter) and electron-microscope (electron microscope EM400-Philips) analyses. The density (d) of the samples was measured by a hydrostatic method using toluene as immersion fluid and the microhardness (HV) was measured by Vickers' method. A thermograph (DTA) was used to record the thermal characteristics of the samples. The module of elasticity (E), the minimum volume of

micro-voids (V_h), the energy of their creation and the compactness (C) were calculated by the equations 1 and 2 [8].

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

$$C = d \cdot \left\{ \sum_i \frac{M_i x_i}{d_i} - \sum_i \frac{M_i x_i}{d} \right\} \left[\sum_i M_i x_i \right]^{-1}, \quad (2)$$

where T_g , T_{cr} and T_m – temperatures of glass transition, crystallization and complete melting; M_i and x_i – molar weight and fraction of the i^{th} component.

3. Results and discussions

The synthesized bulk samples of $Ag_2Te-As_2Se_3-CdTe$ system are with a strong lustre.

The results from the X-ray diffraction show that several compositions are typical glasses without peaks on the diffractograms – these compositions form the glass-forming region (for example p. 6). The compositions on the region boundary (p. 2, p. 8, p. 15, p. 18 and p. 19) show diffraction peaks with low intensity. The diffractograms of the samples outside the glass-forming region (for example p. 9) show strong peaks, these samples are crystalline Fig. 1.

The electronic microscopic observation shows a smooth and homogeneous surface of the samples from the glass-forming region; small crystalline regions are observed on the surface of the samples on the region boundary (crystalline phase); the surface of the samples outside of glass-forming region is rough and there are no regions on it belonging to a glassy phase.

The analysis of the scheme diffractograms Fig. 2 of the compositions lying on the boundary of the glass formation area shows the presence of reflexes of Ag_2Te and As_2Se_3 (p. 2) and of $\text{Ag}_2\text{Te}+\text{CdTe}$ (p. 8, p. 15). In case of higher As_2Se_3 content (p. 18 and p. 19) decomposition of As_2Se_3 is observed in compliance with the scheme: $\text{As}_2\text{Se}_3=2\text{AsSe}+\text{Se}$, the evidence for which is the appearance of the reflexes of AsSe and Se , together with those of Ag_2Te and CdTe .

Based on the syntheses carried out and the results obtained from the visual X-ray diffraction analysis and the electron microscopic analysis we outlined the region of glass-formation in the three-component system $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3-\text{CdTe}$, where $x+y+z=100$ and $m=y/(x+y)$ – Fig. 3. It is extended to the area rich in As_2Se_3 and partially lies on the faces $\text{Ag}_2\text{Te}-\text{As}_2\text{Se}_3$ (50-100 mol% As_2Se_3) and $\text{As}_2\text{Se}_3-\text{CdTe}$ (0-15 mol% CdTe) of the Gibbs' concentration triangle. No glasses were obtained in the system $\text{Ag}_2\text{Te}-\text{CdTe}$. The maximum solubility of CdTe in glasses is ~ 15 mol % [7].

The investigated thermal characteristics of the samples T_g , T_{cr} and T_m , determined by DTA analysis are summarized in Table 2 and typical DTA-curves are shown on Fig. 4.

The introduction of Ag_2Te or CdTe decreases almost in equal degree T_g , because their presence results in growth of the metal component of the chemical bond in the chalcogenide glasses.

The variation of the chemical compound composition affects also slightly the crystallization temperature (T_{cr}), which shows that for all examined compositions one and the same phase crystallizes. The temperature of complete melting (T_m) practically does not depend both on the As_2Se_3 content and on that of Ag_2Te and $CdTe$.

The measured density values (d) of the investigated glasses are in the range 4.8-5.6 $g \cdot cm^{-3}$. At increase of the Ag_2Te content in the composition of the chalcogenide glasses at $z=const$ and increase of the $CdTe$ content at $m=const$, d grows, because $d_{Ag_2Te} > d_{As_2Se_3}$ and $d_{CdTe} > d_{As_2Se_3}$ ($d_{Ag_2Te} = 8.5$; $d_{CdTe} = 5.86$ и $d_{As_2Se_3} = 4.75$ $g \cdot cm^{-3}$).

The visible deviation of the dependences $d(m)$ and $d(z)$ from the additive law is an indication for the chemical interaction between Ag_2Te and $CdTe$ on the one hand and As_2Se_3 on the other, i.e the first two compounds are embedded in the structure carcass of the glass and not dispersed in it as second (third) phase.

The measured microhardness values (HV) of the investigated glasses are in the range 57-85 $kgf \cdot mm^{-2}$. In case of increase of Ag_2Te content (at $z=const$), as well as decrease of $CdTe$ content (at $m=const$), HV decreases as $HV_{As_2Se_3} > HV_{Ag_2Te}$ (respectively 150 and 38 $kgf \cdot mm^{-2}$) and $HV_{CdTe} > HV_{Ag_2Te}$ ($HV_{CdTe} = 60$ $kgf \cdot mm^{-2}$) –

Table. 3.

The elasticity modulus precisely reproduces the micro hardness of the glass and the dependences on the glass compositions $E=f(m)$, at $z=const$ and $E=f(z)$, at $m=const$ are interpreted in analogy with the dependences $HV=f(m)$, at $z=const$ and $HV=f(z)$, at $m=const$.

The minimum volume of the micro voids V_h varies in the range $(8,9-12,5)10^{-3} \text{ nm}^3$ and depends on the glass composition Table 3. The dependence $V_h(m)$ at $z=\text{const}$ passes through a flowing minimum at $m \approx 0,8$. It is logical to assume that at $m > 0,8$ the effect of the glass formative As_2Se_3 is dominant as a result of which more and more distinctly the typical for the vitreous condition properties are revealed: the specimens become more loose (V_h grows), the density d and compactness C - decrease, T_g - increases, the structure becomes more stable (E_h grows), etc. On the other hand, Ag_2Te introduction in the chalcogenide glass (at $z=\text{const}$) also results in V_h increase because the chemical bonds break and the silver atoms and fragments (Ag-Te-Ag) close the broken chains as a result of which the structure network decreases and the disorder in the system grows. As a result of running of the above listed processes the energy for the formation of these micro voids E_h , as well as for the compactness C also decreases with the increase of the Ag_2Te content in the chalcogenide glasses.

The effect of the third component CdTe on V_h and E_h is interesting. The structure unit of CdTe is $\text{CdTe}_{2/2}$. It has linear character and connects the broken chains in the chalcogenide glass while the structure unit of Ag_2Te ($\text{AgTe}_{1/2}$) breaks the long chalcogenide chains and closes them. As a result of this process the minimum volume of the micro voids V_h diminishes. As higher is the CdTe content in the chalcogenide glasses so more stable becomes its structure and the energy necessary for the run of this process E_h decreases. The decrease of V_h in the chemical compounds leads to compression and its compactness C increases.

The properties of the chalcogenide glasses are closely related to the overall mean bond energy which represents a function of the mean coordination number, the type and energy of the chemical bonds between atoms forming the glass.

We determined the correlation between the glass transition temperature T_g , the coordination numbers of glasses $\langle Z \rangle$ and the overall mean bond energy $\langle E \rangle$ in the covalent network of glassy samples of the system $\text{Ag}_x\text{As}_y\text{Cd}_z\text{Se}_m\text{Te}_n$ where x , y , z , n and m are in mol% ($x+y+z+n+m=1$).

We calculated the mean coordination number of glasses $\langle Z \rangle$ by the equation (4) suggested by Tanaka [9] using the coordination numbers $Z_{\text{Ag}}=2$ [10], $Z_{\text{As}}=3$ [11], $Z_{\text{Cd}}=4$ [12], $Z_{\text{Se}}=2$ [11] and $Z_{\text{Te}}=2$ [13].

$$\langle Z \rangle = x.Z_{\text{Ag}} + y.Z_{\text{As}} + z.Z_{\text{Cd}} + m.Z_{\text{Se}} + n.Z_{\text{Te}},$$

(3)

The bond energies of heteropolar bonds (E_{AB}) can be calculated using Paulig's relation [14]:

$$E_{\text{AB}} = 0.5(E_{\text{AA}} + E_{\text{BB}}) + 23.(X_{\text{A}} - X_{\text{B}})^2,$$

(4)

where E_{AA} and E_{BB} - homopolar bond energies for A and B atoms and X_{A} and X_{B} - their electronegativity.

We determined the overall mean bond energy $\langle E \rangle$ with the Tichy's equation [11] of complex chalcogenide systems:

$$\langle E \rangle = \bar{E}_c + \bar{E}_{rm},$$

(5)

where: \bar{E}_c - the average energy of cross-linking/atom calculated with the equation (6);

\bar{E}_{rm} - the average bond energy per atom of the “remaining matrix”.

$$\bar{E}_c = P_p \cdot E_{hb},$$

(6)

$$\bar{E}_{rm} = \frac{2(0.5 \langle Z \rangle) \cdot E_{\Delta}}{\langle Z \rangle} \text{ at } R < 1,$$

(7)

where: E_{hb} is the average heteropolar bond energy for glasses with composition

$A_x B_y C_z D_m E_n$. It is calculated with the equation (8):

$$E_{hb} = \frac{(x \cdot Z_A \cdot E_{A-D} + y \cdot Z_B \cdot E_{B-D} + z \cdot Z_C \cdot E_{C-D} + x \cdot Z_A \cdot E_{A-E} + y \cdot Z_B \cdot E_{B-E} + z \cdot Z_C \cdot E_{C-E})}{(x \cdot Z_A + y \cdot Z_B + z \cdot Z_C)}, \quad (8)$$

where: E_{A-D} , E_{B-D} , E_{C-D} , E_{A-E} , E_{B-E} , and E_{C-E} are the heteropolar bond energies Ag-Se, As-Se, Cd-Se, Ag-Te, As-Te, Cd-Te – Table 4.

We calculated the R coefficient determining the content of chalcogen in glasses with the equation (9):

$$R = \frac{m.Z_D + n.Z_E}{(x.Z_A + y.Z_B + z.Z_C)} \quad (9)$$

When $R > 1$ the system is rich in chalcogen and has heteropolar bonds and chalcogen-chalcogen bonds.

When $R = 1$ the system has a stoichiometric composition because it has heteropolar bonds only.

When $R < 1$ the system is deficient in chalcogen and has heteropolar bonds and metal-metal bonds.

The studied system is deficient in chalcogen because $R < 1$ [$R = 0.8872$].

In this case the degree of cross-linking/atom P_p (for $R < 1$) is calculated with the equation (10):

$$P_p = \frac{m.Z_{Se} + n.Z_{Te}}{(x + y + z + m + n)}, \quad (10)$$

The average bond energy per atom of the “remaining matrix” \bar{E}_{rm} (when $R < 1$) is calculated by an equation (7).

In the equation (11) E_{A-A} , E_{B-B} , E_{C-C} , E_{A-B} , E_{B-C} and E_{A-C} are respectively the bond energies Ag-Ag, As-As, Cd-Cd, Ag-As, As-Cd and Ag-Cd – Table 4.

$$E_{\langle \rangle} = \frac{(E_{A-A} + E_{B-B} + E_{C-C} + E_{A-B} + E_{B-C} + E_{A-C})}{6}.$$

(11)

The values for $\langle Z \rangle$, \bar{E}_c , \bar{E}_{rm} and $\langle E \rangle$ are summarized in Table 5.

The values of T_g according to $\langle E \rangle$ [actually from $(0.38 \langle E \rangle - 0.47)$] are shown on Fig. 5.

A good correlation between T_g and $(0.38 \langle E \rangle - 0.47)$ is observed. This dependence has a dimension not worse than $\pm 0.1 T_g$ and it can be explained very well with Arrhenius relation for viscosity (μ), given in the following type:

$$\mu(T_g) = \mu_0 \exp\left(\frac{E_\mu}{kT_g}\right),$$

(12)

This dependence is suggested by Tanaka [9], who accepts that $\mu(T_g) = 10^{13}$ Poise and $\mu_0 = 10^0 - 10^{-5}$ Poise.

Having in mind $\mu(T_g) = 10^{13}$ Poise and $\mu_0 = 10^{-3}$ Poise, for $T_g = f(E_\mu)$ is received the expression:

$$T_g = 314.E_\mu, \quad (13)$$

The experimental values of T_g and $\langle E \rangle$ follow the correlation:

$$T_g = 119.68 \langle E \rangle - 143.39 \quad (14)$$

By equation 13 and 14 E_μ is defined:

$$E_\mu = 0.38 \langle E \rangle - 0.46 \quad (15)$$

By analogous way A and B constants in the correlation are determined:

$$T_g = A(0.38 \langle E \rangle - 0.46) - B \quad (16)$$

$$T_g = 318.62(0.38 \langle E \rangle - 0.46) - 2.9238 \quad (17)$$

$$T_g = 318.44(0.38 \langle E \rangle - 0.47)$$

(18)

The obtained values of the line slope (318.44 K.eV^{-1}) correspond exactly to the theoretically planned values (314 K.eV^{-1}), which is calculated through Tanaka's model.

A correlation was established between the physico-chemical properties and the composition of glasses

4. Conclusion

(i) For the first time the area of glass formation in the three component system $\text{Ag}_2\text{Te-As}_2\text{Se}_3\text{-CdTe}$ is outlined.

(ii) There are defined basic physical-chemical characteristics of the obtained chalcogenide glasses: density (d), micro hardness (HV) and compactness (C); softening temperature (T_g), crystallization temperature (T_{cr}) and melting temperature (T_m); elasticity modulus (E), volume of micro voids (V_h) and energy of micro voids formation (E_h), dependences between them and the composition being found out.

(iii) The full average energy of the chemical bonds $\langle E \rangle$ is calculated and correlation of the type $T_g=318.44(0.38\langle E \rangle-0.47)$ is found out that is in compliance with the Tanaka's model [9].

* * *

Acknowledgments. The authors acknowledge thankfully the financial support for this work from the Ministry of education and science (Fond “Scientific investigations”- contract TN-1503/05).

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Table. 1. Composition and state of the investigated samples from the $(\text{Ag}_2\text{Te})_x(\text{As}_2\text{Se}_3)_y(\text{CdTe})_z$ system.

sample №	composition, mol %			state
	x	y	z	
p. 1	55.0	45.0	0	crystal
p. 2	50.0	50.0	0	glass + crystal
p. 3	40.0	60.0	0	glass
p. 4	30.0	70.0	0	glass
p. 5	20.0	80.0	0	glass
p. 6	10.0	90.0	0	glass
p. 7	40.5	49.5	10.0	crystal
p. 8	36.0	54.0	10.0	glass + crystal
p. 9	27.0	63.0	10.0	glass
p. 10	18.0	72.0	10.0	glass
p. 11	9.0	81.0	10.0	glass
p. 12*	0	90.0	10.0	glass
p. 13*	0	85.0	15.0	glass + crystal
p. 14	32.0	48.0	20.0	crystal
p. 15	24.0	56.0	20.0	glass + crystal
p. 16	16.0	64.0	20.0	glass
p. 17	8.0	72.0	20.0	glass

p. 18	15.0	60.0	25.0	glass + crystal
p. 19	7.5	67.5	25.0	glass + crystal
p. 20	22.5	52.5	25.0	crystal

* - № 12, 13 are studied in our previously work – [7]

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Table 2. Thermal properties of the samples from the $(\text{Ag}_2\text{Te})_x(\text{As}_2\text{Se}_3)_y(\text{CdTe})_z$ system, where $m=y/(x+y)$

sample №	composition, mol %			m	T_g , °C	T_{cr} , °C	T_m , °C
	x	y	z				
p. 3	40	60	0	0.6	141	199	328
p. 4	30	70	0	0.7	155	203	324
p. 5	20	80	0	0.8	165	210	328
p. 6	10	90	0	0.9	189	212	328
p. 9	27	63	10	0.7	145	208	330
p. 10	18	72	10	0.8	150	210	333
p. 11	9	81	10	0.9	174	220	328
p. 16	16	64	20	0.8	138	217	325
p. 17	8	72	20	0.9	150	224	326

Table 3 Physical and thermomechanical properties of the samples from the
 $(Ag_2Te)_x(As_2Se_3)_y(CdTe)_z$ system, where $m=y/(x+y)$.

sample №	composition,			m	d, g.cm ⁻³	HV, kgf.mm ⁻²	E, kgf.mm ⁻²	V _h , 10 ⁻³ nm ³	E _h , kJ.mol ⁻¹	C -
	mol %									
	x	y	z							
p. 3	40	60	0	0.6	5.60	57	850	12.5	4.33	-0.014
p. 4	30	70	0	0.7	5.42	64	960	12.2	4.76	0.002
p. 5	20	80	0	0.8	5.18	70	1056	11.8	5.07	0.003
p. 6	10	90	0	0.9	4.82	79	1190	12.0	5.80	-0.076
p. 9	27	63	10	0.7	5.60	68	1022	10.7	4.46	0.030
p. 10	18	72	10	0.8	5.40	74	1118	10.1	4.61	0.038
p. 11	9	81	10	0.9	5.06	82	1229	10.7	5.35	0.013
p. 16	16	64	20	0.8	5.50	78	1166	8.9	4.24	0.048
p. 17	8	72	20	0.9	5.22	85	1272	8.9	4.61	0.033

Table 4. Bond energy of the glasses from the $(\text{Ag}_2\text{Te})_x(\text{As}_2\text{Se}_3)_y(\text{CdTe})_z$ system.

Bond	E, eV	Reference	Bond	E, eV	Reference
Ag-Ag	1.69	[15]	Ag-Se	2.10	[18]
As-As	2.07	[16]	Ag-Te	2.03	[18]
Cd-Cd	7.36	[17]	As-Cd	1.94	[14]
Se-Se	2.14	[16]	As-Se	2.26	[16]
Te-Te	1.65	[16]	As-Te	1.99	[14]
Ag-As	2.17	[14]	Cd-Se	1.32	[19]
Ag-Cd	4.54	[14]	Cd-Te	1.04	[19]

Table 5. Mean coordination number of glasses $\langle Z \rangle$, overall mean bond energy $\langle E \rangle$, \bar{E}_c and \bar{E}_{rm} of samples from the $Ag_xAs_yCd_zSe_mTe_n$ system.

sample №	composition, mol %					$\langle Z \rangle$	\bar{E}_c , eV	\bar{E}_{rm} , eV	$\langle E \rangle$, eV
	x	y	z	m	n				
p. 3	19	29	0	43	9	2.29	4.38	0.30	4.68
p. 4	13	32	0	48	7	2.32	4.65	0.17	4.82
p. 5	9	35	0	52	4	2.35	4.74	0.15	4.89
p. 6	4	38	0	56	2	2.38	4.92	0.08	5.00
p. 9	13	30	2	45	8	2.30	4.35	0.26	4.61
p. 10	8	33	2	50	7	2.37	4.69	0.13	4.81
p. 11	4	36	2	54	4	2.40	4.78	0.11	4.89
p. 16	8	31	5	47	9	2.41	4.42	0.23	4.65
p. 17	4	34	5	50	7	2.44	4.51	0.22	4.72

Figure 1:

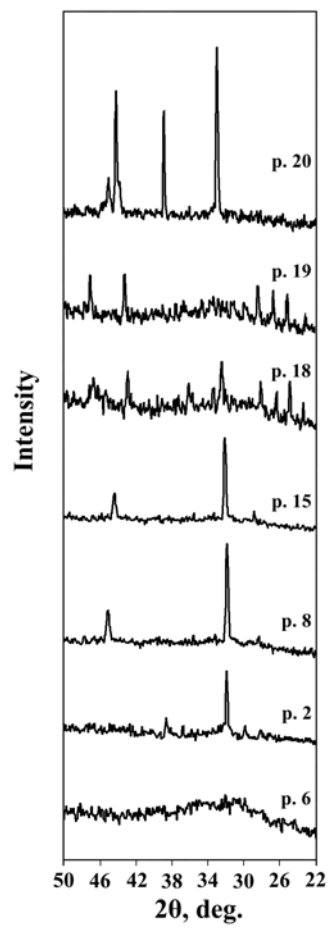


Figure 2:

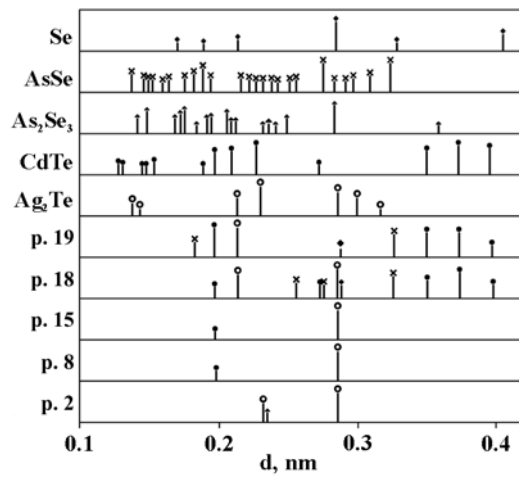


Figure 3:

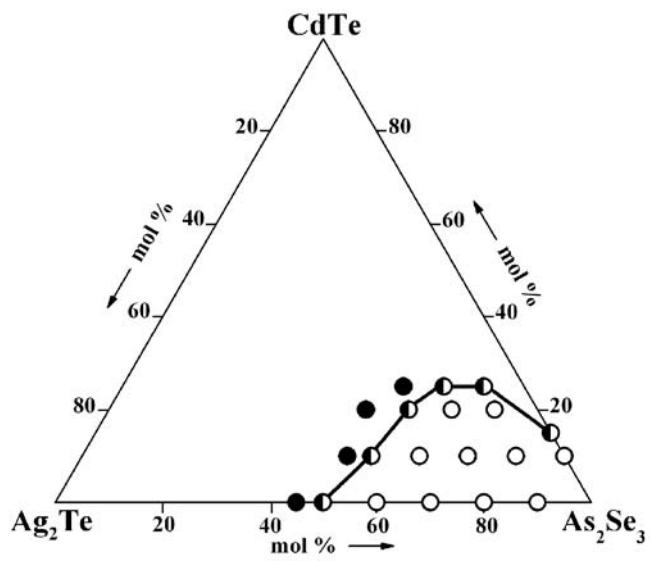


Figure 4:

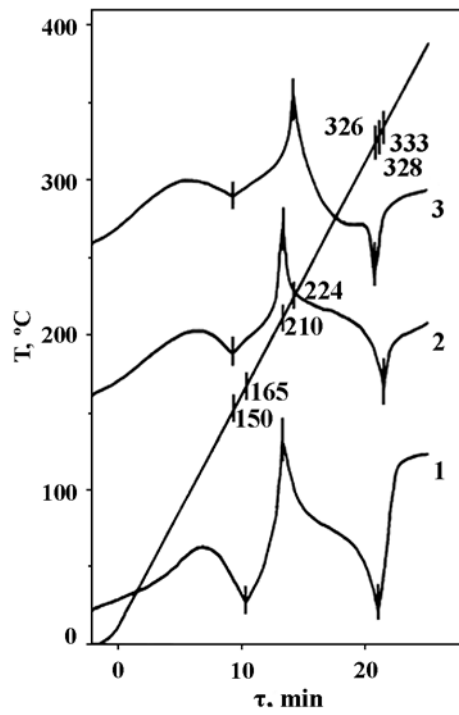
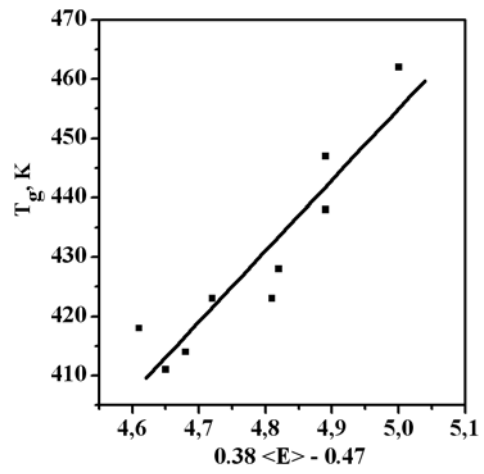


Figure 5:



Accepted manuscript