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Raman scattering and X-ray diffraction studies of $Ge_{16}Sb_{24}Se_{60}$ alloys

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Abstract. The structure and vibrational properties of glassy $Ge_{16}Sb_{24}Se_{60}$ alloys were studied using X-ray diffraction and Raman scattering. The experimental X-ray diffraction patterns confirmed the amorphous nature of the obtained alloys. The latter were used for calculating the radial distribution functions. These calculations gave the positions of the nearest-neighbour peak r_1 – 2.56 Å and the second nearest-neighbour peak r_2 – 3.79 Å. The obtained r_1 value aligns well with the known Ge-Se and Sb-Se bond lengths from the literature. Similar r_1 value was also observed for Ge-Sb-Se glasses of different compositions. The r_2/r_1 ratio of 1.48 yields the value of the bond angle $\theta = 95.5°$. The observed bands in the Raman spectra of the studied $Ge_{16}Sb_{24}Se_{60}$ samples show that these glasses contain different nanophases. The Raman spectra can be interpreted in terms of vibrational modes of Ge-Se and Sb-Se binary glasses and films.

Keywords: X-ray diffraction, Raman spectroscopy, glassy Ge-Sb-Se alloys.

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1. Introduction

Chalcogenide glasses have garnered extensive research interest due to their exceptional structural, optical, and electronic properties. These materials exhibit a remarkable combination of mid-infrared (MIR) transparency, high refractive index, and significant optical nonlinearity, making them highly promising for applications in infrared optics, fibre optics, integrated optics, nonlinear photonics, telecommunications systems, and optoelectronics [1–5]. One of the distinctive features of chalcogenide glasses is their ability to photo-structural transformations, which are accompanied by changes in optical and chemical properties [1, 2]. These include photo-induced effects, polarization-dependent structural modifications, localized expansion or contraction, and photoinduced dichroism.

The properties of chalcogenide glasses can be tailored for specific applications through compositional variation, doping, and the fabrication of nanocomposites [6–10]. Nanocomposite materials derived from chalcogenide glasses enable direct recording of surface reliefs, facilitated by polarization-dependent processes [7–9]. Ge-Sb-Se alloys exhibit a broad glass formation region (Fig. 1 [11]), allowing for customizable properties through compositional adjustments [12–16]. These alloys are characterized by high nonlinear optical properties, excellent transmission in the IR, and notable acousto-optic properties [17–25]. Such features allow for targeted optimization of the material parameters, making them suitable for diverse applications, including optical sensors, infrared lenses, amplifiers, and switching components in optical communication systems [17, 18, 21, 26–28].

Ge-Sb-Se alloys, which are part of the chalcogenide family, exhibit unique bonding characteristics. Particularly, the resonant bonding involves delocalized electrons that contribute to the material's unique optical and electronic properties. While resonant bonding is a feature often associated with materials like Ge-Sb-Te alloys [27], it is less commonly discussed in the context of Ge-Sb-Se alloys [28].

Moreover, Ge-Sb-Se alloys exhibit structural phase transitions, influencing their thermal stability, mechanical strength, and optical performance [29]. Structural analyses using diffraction and extended X-ray absorption fine structure (EXAFS) techniques [30–32] have demonstrated that the atomic network of Ge-Sb-Se glasses accords with the Chemical Ordered Network Model (CONM [33]). This model suggests a preferential bonding between M and Se atoms (M = Ge or Sb), minimizing M-M and Se-Se bonds. The structural network comprises units, namely $GeSe_{4/2}$ and $SbSe_{3/2}$, which are interconnected *via* their corners, edges, or Se-Se bridges in selenium-rich glasses. In selenium-deficient systems, where only several Se atoms are available to satisfy the bonding requirements of M atoms, the formation of M-M bonds is also anticipated [30, 31].

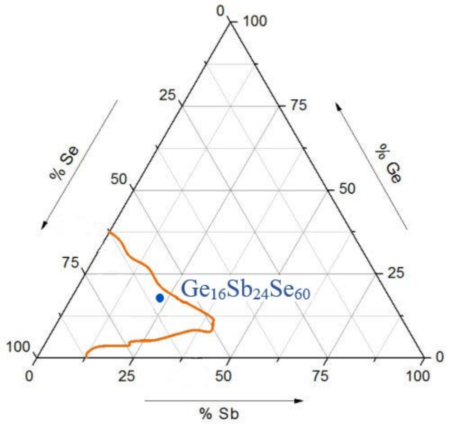

Fig. 1. Glass forming region for Ge-Sb-Se alloys (orange line) according to [11]. ● – studied $Ge_{16}Sb_{24}Se_{60}$ glass.

Diffraction techniques are an efficient method for ascertaining the structure of chalcogenide glasses, particularly their near-order structure [30–32]. Raman spectroscopy serves as a powerful technique for studying the vibrational properties of both crystalline [34] and amorphous materials [35] and is an efficient complementary method to X-ray diffraction.

In this study, the $Ge_{16}Sb_{24}Se_{60}$ alloys were systematically investigated using X-ray diffraction and Raman scattering, providing insights into their structural and vibration characteristics.

2. Experimental details

The studied bulk $Ge_{16}Sb_{24}Se_{60}$ alloys were prepared using the conventional melt quenching technique. Appropriate quantities from high-purity (5 N) elements were sealed into quartz ampoules after evacuation down to a pressure of 10^{-3} Pa. The sealed ampoules were kept inside a rotary furnace at 950 °C, and after homogenization for 24 h, the melts were quenched in air. X-ray diffraction (XRD) patterns of the samples were recorded using a Rigaku SmartLab® X-ray diffractometer with CuKα radiation source ($\lambda = 1.5406$ Å). The diffraction data were collected within the range of 2θ angles 2°…120°, with a 0.05° step.

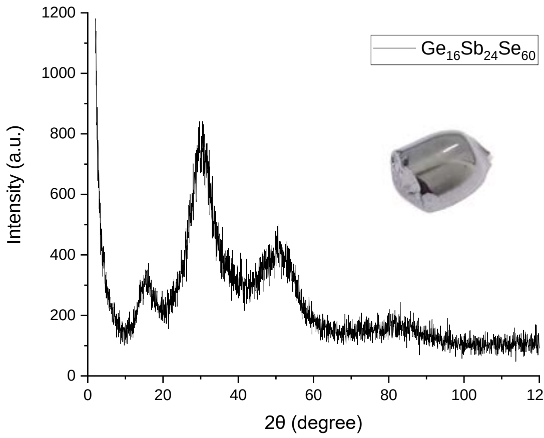

Fig. 2. Diffraction pattern of $Ge_{16}Sb_{24}Se_{60}$ glass. Insert – photo of $Ge_{16}Sb_{24}Se_{60}$ glass ingot.

All X-ray experiments were performed at ambient temperature. The XRD patterns of the studied Ge-Sb-Se alloys confirmed the amorphous nature of the samples (Fig. 2) and were analyzed using the Rad GTK+ software.

Radial distribution function (RDF(r)) is defined as the number of atoms lying at distances within the range (r, $r + dr$) from the centre of an arbitrary atom and is written as follows:

$$\mathrm{RDF}(r) = 4\pi r^2 \rho(r). \qquad (1)$$

Here, $\rho(r)$ is the density function, which represents an atomic pair correlation function. The average coordination number, N, in a spherical shell between the radii r_1 and r_2 around any given atom can be calculated as the number of atoms in the area between r_0 and r, where r_0 is the first minimum of $4\pi r^2\rho(r)$. In other words, r_0 is the lower limit of r, below which $\rho(r)$ is equal to zero. The position of the first peak in the radial distribution function gives the nearest-neighbour bond length r_1, and similarly, the position of the second peak gives the next-neighbour distance r_2. The RDF yields only a limited amount of information, restricted essentially to the local structure around a given atom, *i.e.*, the bond lengths and bond angles. Using both the bond lengths r_1 and r_2, one can calculate the value of the bond angle θ given by [32]:

$$\theta = 2\sin^{-1}(r_2/2r_1). \qquad (2)$$

Raman spectra were acquired with a LabRAM HR setup from HORIBA Scientific. Measurements were performed in a backscattering geometry at room temperature using a $\lambda = 514.7$ nm laser with a power of 70 μW, and a 50× objective for excitation. The measurement range was from 50 to 1500 cm^{-1}, with a resolution of 1 cm^{-1} and an accumulation time of 120 s. The obtained Raman spectra were analyzed using CoRa software [36].

3. Results and discussion

3.1. X-ray diffraction data

The experimental X-ray diffraction patterns were used for calculating the radial distribution function (Fig. 3). The short-range parameters are the number of the nearest neighbouring atoms (coordination number), their type, the distance from them to the central atom (the radius r_1 of the first coordination sphere), and angular distribution of the atoms concerning the central atom, which is determined by the chemical bond angles (valence angles φ). This definition limits short-range order to the first coordination sphere. At the same time, the short-range parameters define not only the first, but also, at least partially, the second coordination sphere. As can be seen from the expression (2), the radius of the second coordination sphere r_2 is defined by the radius of the first coordination sphere and the bond angle.

Table 1. Short-range parameters of the $Ge_{16}Sb_{24}Se_{60}$ glasses.

Composition	r_1, Å	r_2, Å	r_2/r_1	θ, degree
$Ge_{16}Sb_{24}Se_{60}$	2.56	3.79	1.48	95.5°

The obtained radial distribution function (Fig. 3) gives the following values of the radii of the first and second coordination spheres, and using the r_2/r_1 value of 1.48, the bond angle θ was calculated (Table 1).

According to the works [30, 31], the bond lengths in Ge-Sb-Se glasses are within the following limits: Ge-Se – 2.36…2.37 Å, Ge-Ge – 2.45…2.46 Å, Ge-Sb – 2.65 Å, Sb-Se – 2.58…2.59 Å. It is worth noting that the structure of the $Ge_{20}Sb_{20}Se_{60}$ glass (which is quite close in composition to the $Ge_{16}Sb_{24}Se_{60}$ glasses studied in this work) corresponds to the model of a chemical ordered network and that, along with the $GeSe_{4/2}$ and $SbSe_{3/2}$ structural units, $Se_{3/2}Ge\text{-}Ge(Sb)Se_{3/2}$ blocks are present to compensate for the selenium deficiency [30]. Diffraction and extended X-ray absorption fine structure (EXAFS) measurements proved that the structure of Ge-Sb-Se glasses can be described by the CONM [30]. According to the CONM, the Ge-Se, and Sb-Se bonds are preferred. In low-Se systems, where there are not enough Se atoms to meet the binding needs of M atoms (M = Sb, Ge), M-M bonds are also expected [30, 31].

3.2. Raman spectroscopy

The vibrational properties of $Ge_{16}Sb_{24}Se_{60}$ were studied by Raman spectroscopy. The $Ge_xSb_{40-x}Se_{60}$ family belongs to the line joining the non-stoichiometric Ge_2Se_3 and the stoichiometric Sb_2Se_3. Since this tie line is below the stoichiometric one, these glasses are referred to as ‘chalcogen deficient’, *i.e.*, Se-Se bonds are assumed not to exist in $Ge_xSb_{40-x}Se_{60}$ [30]. Schematically, the glass network is considered mainly as a combination of tetrahedral $GeSe_{4/2}$ and pyramidal $SbSe_{3/2}$ structural units. Raman experiments for some compositions of $Ge_xSb_{40-x}Se_{60}$ glasses suggested $GeSe_{4/2}$, and $SbSe_{3/2}$ structural units, as well as some violation of the chemical ordering in the form of Se–Se and M–M bonds [30]. According to previous studies, up to ten bands can be expected in the Raman spectra of Ge-Sb-Se glasses. These bands are attributed to vibrations of the corresponding structural units. The peak positions of these bands are summarized in Table 2 along with their assignment and references.

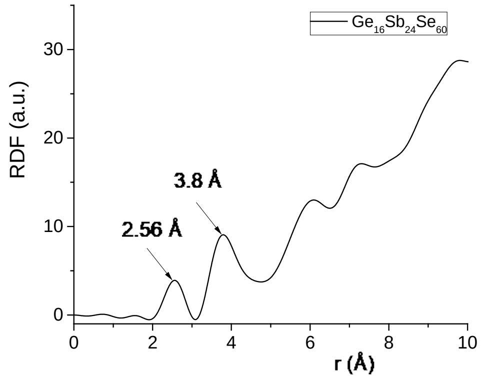

Fig. 3. Radial distribution function of the studied $Ge_{16}Sb_{24}Se_{60}$ glasses.

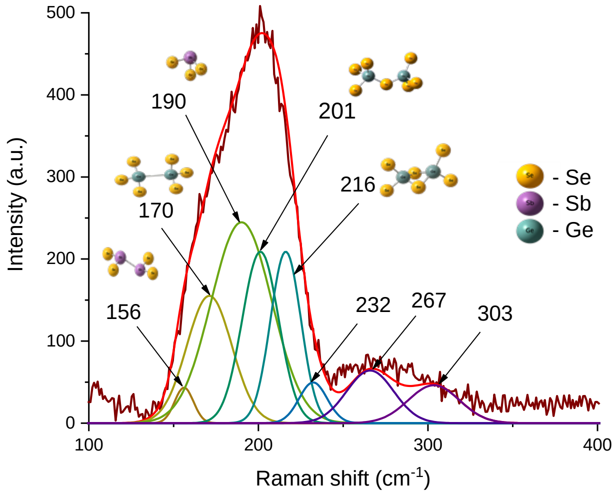

Fig. 4. Raman spectra of $Ge_{16}Sb_{24}Se_{60}$ glass.

The Raman spectrum of $Ge_{16}Sb_{24}Se_{60}$ sample studied in the present work is shown in Fig. 4. We decomposed the broad bands of the Raman spectra into several Gaussian components using a series of Gaussian peaks with widths appropriate for glasses. The peak

Table 2. Assignments of vibrational bands detected in the Raman spectra of Ge-Sb-Se glasses.

Wavenumber, cm^{-1}	Assignment	References
~ 100 cm^{-1}	vibrations in Sb-$Se_{3/2}$ pyramids	[14]
~ 150 cm^{-1}	Sb-Sb vibrations in $Se_2Sb\text{-}SbSe_2$	[14, 29, 37−39]
~ 170 cm^{-1}	Ge-Ge vibrations in Ge-Se_4-nGen	[14, 39]
~ 190 cm^{-1}	Sb-Se vibrations in Sb-$Se_{3/2}$ pyramids	[29, 37, 39]
~ 200 cm^{-1}	symmetric stretching vibrations of corner-shared $GeSe_{4/2}$ tetrahedra	[29, 37−39]
~ 215 cm^{-1}	vibrations of edge-shared $GeSe_{4/2}$ tetrahedra	[14, 37−39]
~ 235–250 cm^{-1}	vibrations of different structural units of Se	[14, 39]
~ 265 cm^{-1}	vibrations of corner-shared tetrahedra of $GeSe_{4/2}$	[14]
~ 285–300 cm^{-1}	asymmetric vibrations of tetrahedra of $GeSe_{4/2}$	[14]
~ 250–330 cm^{-1}	Ge±Ge bonds in modified $Se_3Ge\pm(GeSe_2)_n\pm GeSe_3$ (n = 0:1) or $Ge\pm Ge_mSe_{4\text{-}m}$ (m = 1, 2, 3, 4)	[14]

widths, heights, and center frequencies were optimized to fit the experimental data. The frequency assignments of known structural units in Sb-Se, Ge-Se, and Ge-Sb-Se glasses, summarized in Table 2, were used to perform the peak-fitting analyses and to compare the relative contribution of each structural unit in the spectrum of amorphous $Ge_{16}Sb_{24}Se_{60}$ glasses. The deconvolution of Raman spectra of the studied glasses was carried out using the mentioned bands (150, 170, 190, 200, 215, 265 cm^{-1} [14, 29, 37−39]) and a good match with the experimental spectra was obtained. Consequently, Raman data show the presence of $GeSe_{4/2}$ and $SbSe_{3/2}$ structural units in our $Ge_{16}Sb_{24}Se_{60}$ glasses, as well as some disorder of chemical ordering in the form of structural units with M-M bonds (M = Ge, Sb). However, further studies of their structure at the atomic level are still needed to better understand their structural properties.

4. Conclusions

The structural characteristics of $Ge_{16}Sb_{24}Se_{60}$ glasses were investigated using X-ray diffractometry and Raman spectroscopy. Short-range order parameters were determined and comprehensively analyzed. The findings indicate that the structure of these glasses aligns with the chemically ordered network model, wherein Ge-Se and Sb-Se bonds dominate the bonding configuration. In cases of chalcogen deficiency, M-M bonds (where M represents Ge or Sb) emerge within the network. These results are in agreement with previously reported findings on the structural properties of Ge-Sb-Se glasses.

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Dzhagan V.M.: analysis, writing – review and editing.
Shportko K.V.: analysis, writing – review & editing.
Selyshchev O.: investigation, formal analysis.
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Дослідження сплавів $Ge_{16}Sb_{24}Se_{60}$ методами раманівського розсіяння та рентгенівської дифракції

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Анотація. Структуру та коливні властивості склоподібних сплавів $Ge_{16}Sb_{24}Se_{60}$ досліджено за допомогою рентгенівської дифракції та раманівського розсіяння. Аморфну природу отриманих сплавів підтверджено експериментальними рентгенограмами. Останні були використані для розрахунку функцій радіального розподілу. Такі розрахунки дали положення піка найближчого сусіда r_1 – 2,56 Å та другого піка найближчого сусіда r_2 – 3,79 Å. Отримані значення r_1 добре узгоджуються з відомими з літератури довжинами зв'язків Ge-Se та Sb-Se. Подібні значення r_1 також спостерігалися для стекол Ge-Sb-Se різного складу. Співвідношення $r_2/r_1 = 1,48$ дає значення кута зв'язку $\theta = 95,5°$. Спостережувані смуги в раманівських спектрах досліджуваних зразків $Ge_{16}Sb_{24}Se_{60}$ показують, що такі стекла містять різні нанофази. Спектри комбінаційного розсіювання можна інтерпретувати з точки зору коливальних мод бінарних стекол і плівок Ge-Se та Sb-Se.

Ключові слова: рентгенівська дифракція, раманівська спектроскопія, склоподібні сплави Ge-Sb-Se.