Experimental study of the structure of chalcogenide glassy semiconductors in three-component systems of Ge-As-Se and As-Sb-Se by means of NQR and EPR spectroscopy

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Abstract: The structure of chalcogenide glassy semiconductors in three-component systems of Ge-As-Se and As-Sb-Se has been studied by means of both NQR (nuclear quadrupole resonance) and EPR (electron paramagnetic resonance) spectroscopy. It is investigated that in the glasses of both systems the value of the electric field gradient at the resonating nuclei grows with increasing concentration of the clusters As₂Se₃ and Sb₂Se₃, thereby increasing the NQR resonance frequencies. It appears that for the Ge-As-Se system the structural transition from a two-dimensional to three-dimensional structure occurs at average coordination number \( \bar{r} = 2.45 \). The EPR spectral parameters of glasses depend on the composition, the average coordination number and the temperature, and these are discussed. The effect of "ageing" for CGS (chalcogenide glassy semiconductors) of As-Sb-Se system due to partial crystallization of the sample is observed from the EPR spectra.

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1. Introduction

In recent years, non-crystalline solids have become of interest for physicists working in both fundamental research as well as applied research fields. On the one hand such
systems are characterized by the lack of long-range order, i.e. strict periodicity of the arrangement of atoms in a microscopic volume. On the other hand they are characterized by the presence of short-range order, i.e., ordered distribution of the nearest neighbours for each atom. The structure, and the chemical and thermodynamic properties of amorphous solids are more difficult to describe in comparison to crystalline and liquid states. As a result, there are still many open questions about the properties of non-crystalline solids.

For this study the chalcogenide semiconductors of ternary Ge-As-Se and As-Sb-Se systems (the composition of which can be changed in the synthesis process) have been chosen from the large number of disordered chalcogenide materials of poorly understood inorganic origin. A characteristic feature of chalcogenide glasses is their ability to deviate significantly from the stoichiometric composition, i.e. from the respective chemical formula. For elements of a given system, it is usually impossible to obtain glass of arbitrary composition, i.e. during vitrification there are certain limits within which the amorphous materials may be prepared by rapid cooling of the melt. At the same time, samples of compositions located outside the glass forming region can be obtained, similar to production of amorphous silicon by deposition from the uncondensed state. Glassy chalcogenide compounds are more thermodynamically stable compared to amorphous silicon and germanium. Therefore, annealing usually does not lead to substantial changes in physical properties.

Nuclear quadrupole resonance (NQR) is one of the most sensitive methods to study the local structure of the material, the electron density distribution near the nucleus, the nature of the defects, the mobility of individual molecules and groups of atoms in the molecule, also. NQR is used to study the physical properties of solids (molecular crystals, polymers, metals, glass, vitreous semiconductors). Applications of NQR in the study of crystals, particularly semiconductors, are based on the relationship between the structure of crystals and crystal-field gradients. In contrast to nuclear magnetic resonance, the NQR resonance frequencies are directly determined by the crystalline structure. Application of the new research methods in NQR spectroscopy (creation of the theory of the echo signal for very broad NQR lines [1] and the geometric phase of the signal in the NQR experiments [2]) to determine the asymmetry of the electric field gradient tensor (EFG) in disordered structures allowed us to obtain more information on the structure of compounds of this type. The NQR studies have been accompanied by the electron paramagnetic resonance (EPR) study which provides information about the short-range order of the atomic arrangement. Until now nuclear quadrupole resonance spectroscopy has not been widely used to study the structure of disordered solids [3–7]. Another radiospectroscopic method, the method of electron paramagnetic resonance spectroscopy, is used for this purpose more often [8]. These two methods provide information about the local structure, and the atomic order in amorphous solids.

The purpose of this paper is to study the structure of chalcogenide semiconductors of ternary Ge-As-Se and As-Sb-Se systems by the methods of NQR and EPR spectroscopy, to obtain information about the glassy state of matter and its dependence on the composition of the EPR (chalcogenide glassy semiconductors), information about the local mutual coordination atoms, and about the ways of combining structural units providing medium and long-range chemical order.

2. The previous NQR and EPR study of Ge-As-Se and As-Sb-Se glassy semiconductors

The glasses of the Ge-As-Se system exist in the wide glass forming region (to 40% Ge and 70% As) with the average coordination number from $\bar{r} = 2$ for pure selenium up to $\bar{r} = 3.3$ for compounds with high concentration of As and of Ge. It is convenient to consider the local structure of a three-component system based on structure elements of corresponding Ge-Se and As-Se binary systems, since the selenium is the main component of glass in these semiconductors. According to the model of the formal valence environment, the atoms with a large number of valence electrons form a sublattice of anions. So, the system Ge$_x$As$_y$Se$_{1-x-y}$ has Ge-Se and As-Se bonds. In the selenium rich area, Ge and As atoms form tetrahedral and pyramidal structural cells, respectively, in which branching and the formation of intermolecular bonds between chains of Se atoms are made. If the compounds of Ge-As-Se are rich in Se atoms or in Ge atoms, associated with As atoms, then $\bar{r}$ increases and this process is probably accompanied by forming Ge-Ge and As-As bonds, i.e. leading to effects of the formation of clusters.

The $^{75}$As NQR spectra and their frequencies and linewidths for chalcogenide semiconductors of Ge-As-Se different compositions are presented in our previous work [9]. The results of our study of the NQR spectra for the samples of this system show that changing their chemical composition leads to significant differences in the NQR spectra. Study of the dependence of internal friction in the Ge-As-Se glasses on the average coordination number in work [10] has concluded that as $\bar{r}$ increases to a value of 2.27–2.56, the transition occurs from a one-dimensional to a two-dimensional structure such
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3. Experiment

The process of obtaining samples of CGS for the study (Table 1) is described in [17].

To describe the structure of amorphous solids, the average coordination number which is a measure of the short-range order, is typically used. The average coordination number of Ge-As-Se (Ge,As$_2$Se$_{1-x-y}$) and As-Sb-Se (As,Sb$_2$Se$_{1-x-y}$) glasses has been calculated by the equation from [18], where the coordination numbers for Ge, As, Sb and Se are 4, 3, 3 and 2 respectively.

NQR experiments have been carried out using a pulsed FT-NQR spectrometer type NQS-300 from MBC ELECTRONICS Company. A two-pulse Hahn spin-echo pulse sequence $\frac{\pi}{2}$ – $\tau$ – $\pi$ was used for obtaining a NQR line. For all the samples the duration of the first pulse ($\frac{\pi}{2}$) was 5 $\mu$s, the interval $\tau$ between pulses was 60 $\mu$s, the duration of the second pulse ($\pi$) was 10 $\mu$s, the repetition period was 150 ms. The sampling period was 0.1 $\mu$s and the number of accumulations used was equal to 1000 – 2000. Measurements have been performed on $^{75}$As isotope at 77 K. To record very wide NQR lines the spectrometer frequency has been varied with steps of 200 kHz. A very wide NQR spectrum is a characteristic feature of the vitreous semiconductor studied here. The NQR line width is about 20 MHz. The reconstruction of the spectra has been carried out on the basis of integral intensities of spin-echo signals. Integrated intensity of the signal at each point was calculated using both the real and imaginary signal components in Matlab software. After that, all the spectra were fitted using a Lorentz line shape in Origin software.

The expression for the sum of the two lines was used for the Ge-As-Se system, because the NQR signal for this system is observed on the nuclei of two atoms of arsenic $^{75}$As. For the As-Sb-Se system the expression for the sum of five lines was used, as for this system the NQR signal was observed, presumably, at the same time on one antimony atom nuclei $^{121}$Sb, two atoms $^{123}$Sb, and two arsenic atoms $^{75}$As.

The EPR spectra were observed with a Bruker EPR spectrometer, EMX type, working at 9 GHz frequency. The measurements were performed at 300 K and 77 K temperatures. To calculate the concentration of paramagnetic centres in investigated samples the weak pitch standard sample from Bruker was used with a concentration of $10^{13}$ spins per cm$^3$.

4. The interpretation of the experimental results of the CGS study and their discussion

The interpretation of the experimental results of the CGS research is carried out on the basis of assumptions about clustering structure, as discussed above. Publications [9, 10] include the NQR spectra and spectral parameters of $^{75}$As, $^{121}$Sb and $^{123}$Sb (T = 77 K) glassy systems of Ge-As-Se and As-Sb-Se in the frequency range of 48-70 MHz. For the As-Sb-Se system, the NQR spectra indicate that besides the lines from the two nonequivalent positions of arsenic $^{75}$As nuclei, there are also the lines observed from the nuclei $^{121}$Sb and $^{123}$Sb, which complicates the NQR spectrum itself and its interpretation. Based on the
Table 1. Investigated samples of chalcogenide glassy semiconductors: chemical formula (the ratio of concentrations), chemical formula (atomic %) and average coordination number of the samples.

| Nr | Chemical formula (the ratio of concentrations) | Chemical formula (atomic %) | \( \bar{r} \) |
|----|-----------------------------------------------|-----------------------------|------------|
| 1  | (As\(_2\)Se\(_3\))\(_{0.9}\)(GeSe\(_2\))\(_{0.1}\) | Ge\(_{0.021}\)As\(_{0.375}\)Se\(_{0.604}\) | 2.417 |
| 2  | (As\(_2\)Se\(_3\))\(_{0.8}\)(GeSe\(_2\))\(_{0.2}\) | Ge\(_{0.043}\)As\(_{0.348}\)Se\(_{0.609}\) | 2.434 |
| 3  | (As\(_2\)Se\(_3\))\(_{0.7}\)(GeSe\(_2\))\(_{0.3}\) | Ge\(_{0.608}\)As\(_{0.318}\)Se\(_{0.614}\) | 2.454 |

As shown in [17], the asymmetry parameter of the EFG tensor is sensitive to changes in the average coordination number, i.e., to the immediate environment of arsenic. It has been found that function \( \eta(\bar{r}) \), exhibits a maximum at \( \bar{r} = 2.425 \) which probably indicates a change in the glass structure.

Table 2 shows the frequency and width of NQR \(^{75}\)As in Ge-As-Se glasses (\( T = 77 \) K) from the works [6, 9].

Fig. 2 shows the dependence of the \(^{75}\)As NQR frequency on the average coordination number \( \bar{r} \) in a disordered Ge-As-Se system. As it can be seen from the figure, there is a good correlation between the NQR frequency and coordination number (linear dependence). With the increasing \( \bar{r} \) value of the EFG on the nuclei increases, this, in its turn, leads to an increase in the NQR frequencies. In addition CGS are characterized by an average coordination number, which is the radius of the first coordination sphere and is equal to the shortest distance between neighboring atoms. Fig. 3 shows the change in the width of the NQR lines, as a function of the average coordination number of glassy Ge-As-Se. It should be noted that as the line width \( \nu_1 \) increases with average coordination number, the line width \( \nu_2 \), on the contrary, decreases.

Fig. 4 shows the dependence of the integrated intensity of the \(^{75}\)As NQR lines on the average coordination number. The rhombs correspond to the first non-equivalent positions of arsenic in the structural unit of As\(_2\)Se\(_3\), circles correspond to the second non-equivalent positions of arsenic. Figs. 5 and 6 show the dependences of the displacement of the NQR frequencies on the concentration of GeSe\(_2\) and the change in the widths of the NQR lines on the concentration of these clusters. As it can be seen from Fig. 4, the integrated intensities of the NQR lines for
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Table 2. Frequencies and widths of the NQR lines $^{75}$As in Ge-As-Se glasses (T = 77 K) (* it is taken from [6]).

| Glass compositions $^{75}$As$_{0.21}$Se$_{0.60}$ | NQR frequency (linewidth) $^{75}$As in Ge-As-Se glasses (T = 77 K) | $^{75}$As$_{0.43}$As$_{0.375}$Se$_{0.609}$ | $^{75}$As$_{0.43}$As$_{0.38}$Se$_{0.614}$ | $^{75}$As$_{0.608}$As$_{0.218}$Se$_{0.614}$ | $^{75}$As$_{0.222}$As$_{0.222}$Se$_{0.595}$ | $^{75}$As$_{0.33}$As$_{0.12}$Se$_{0.55}$ |
|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| $^{75}$As$_{0.21}$Se$_{0.604}$                | $\nu_1$, MHz ($\Delta \nu_1$, MHz) $^{75}$As$_{0.43}$As$_{0.375}$Se$_{0.609}$ | $\nu_2$, MHz ($\Delta \nu_2$, MHz) $^{75}$As$_{0.43}$As$_{0.38}$Se$_{0.614}$ | $\nu_1$, MHz ($\Delta \nu_1$, MHz) $^{75}$As$_{0.608}$As$_{0.218}$Se$_{0.614}$ | $\nu_2$, MHz ($\Delta \nu_2$, MHz) $^{75}$As$_{0.222}$As$_{0.222}$Se$_{0.595}$ | $\nu_1$, MHz ($\Delta \nu_1$, MHz) $^{75}$As$_{0.33}$As$_{0.12}$Se$_{0.55}$ |
| 2.417                                       | 51.5±0.2 (2.1±0.7)                                 | 58.4±0.3 (9.3±1.1)                             | 54.4±0.1 (2.9±0.5)                             | 59.2±0.1 (6.4±0.4)                             | 55.1±0.7 (6.9±2.1)                             | 60.9±0.4 (5.8±1.1)                             |
| 58.4±0.1 (9.3±1.1)                           | 54.4±0.1 (2.9±0.5)                                 | 59.2±0.1 (6.4±0.4)                             | 55.1±0.7 (6.9±2.1)                             | 60.9±0.4 (5.8±1.1)                             | 55.1±0.7 (6.9±2.1)                             | 60.9±0.4 (5.8±1.1)                             |
| 2.434                                       | 54.4±0.1 (2.9±0.5)                                 | 59.2±0.1 (6.4±0.4)                             | 55.1±0.7 (6.9±2.1)                             | 60.9±0.4 (5.8±1.1)                             | 55.1±0.7 (6.9±2.1)                             | 60.9±0.4 (5.8±1.1)                             |
| 2.454                                       | 55.1±0.7 (6.9±2.1)                                 | 60.9±0.4 (5.8±1.1)                             | 60.9±0.4 (5.8±1.1)                             | 55.1±0.7 (6.9±2.1)                             | 60.9±0.4 (5.8±1.1)                             | 60.9±0.4 (5.8±1.1)                             |
| 2.670                                       | 61.0±0.7 (7.8±2.0)                                 | 71.0±0.7 (14±2.0)                              | 61.0±0.7 (7.8±2.0)                             | 71.0±0.7 (14±2.0)                              | 61.0±0.7 (7.8±2.0)                             | 71.0±0.7 (14±2.0)                              |
| 2.780                                       | 66.0±0.7 (9.5±2.0)                                 | 73.5±0.7 (14±2.0)                              | 66.0±0.7 (9.5±2.0)                             | 73.5±0.7 (14±2.0)                              | 66.0±0.7 (9.5±2.0)                             | 73.5±0.7 (14±2.0)                              |

As a rough analogy with a grid glass crystalline lattice, let us consider the defects and their impact on the NQR spectra. As it is well known, an increasing concentration of the defects in the crystalline lattice leads to the observation of a decrease in intensity of the absorption line broadening NQR. Impurity molecules alter the electric field gradient on the neighbouring nuclei and form local regions of tension. Each impurity molecule causes a shift of the nuclei NQR frequencies in some sphere of radius $r_c$. This is a critical region which means that all the resonating nuclei included in this region will not contribute to the centre of the absorption line, which reduces the intensity of the line. At a certain concentration of impurities the function of the intensity of the NQR lines on the impurity concentration exhibits an inflection point [19], which means that the number of critical spheres has become so large that they began to overlap. This helps to slow down the decline in the intensity of the absorption
The number of GeSe clusters in the neighbourhood of the first non-equivalent position of arsenic is decreasing with the increase of coordination number and increasing near the second unequal position of arsenic. This is because $v_1$-line intensity increases with the average coordination number, and the $v_2$-line intensity decreases. It should also be noted that in both cases the value of the integrated intensity of the lines approaches the same value as the average coordination number approaches $r = 2.45$. The same can be seen in the graph of change in the width of the $^{75}$As NQR lines in the Ge-As-Se glasses vs. the average coordination number (Fig. 3). It could also confirm the assumption of a structural transition at $r = 2.45$, that is the transition of Ge-As-Se from two-dimensional to three-dimensional structure [20].

EPR spectra of chalcogenide glassy compounds of Ge-As-Se system and their spectroscopic parameters are given in our previous work [17]. It can be argued by analyses of EPR spectra and their parameters for the system Ge-As-Se that the samples $(\text{As}_2\text{Se}_3)_{0.9}(\text{GeSe})_{0.1}$ and $(\text{As}_2\text{Se}_3)_{0.7}(\text{GeSe})_{0.3}$ have similar local structures and the sample $(\text{As}_2\text{Se}_3)_{0.8}(\text{GeSe})_{0.2}$, in contrast, has a radically different local environments. Large linewidths of the EPR centres in CGS of Ge-As-Se system for the first two samples indicate that these compositions of CGS paramagnetic centre interacts with a large number of $^{75}$Ge and $^{75}$As nuclei, located at different distances in the first coordination sphere. The changes of the EPR spectrum with growth of the concentration of germanium and reduction of the concentration of arsenic are observed. The changes lead to the dominance of narrow lines. Such changes of the EPR spectrum observed for the $(\text{As}_2\text{Se}_3)_{0.8}(\text{GeSe})_{0.2}$ compound with an average coordination number $r = 2.434$ may indicate a structural change in the glass.

In contrast to the Ge-As-Se system for the studied samples of the As-Sb-Se system, the EPR signal was observed as a single broad isotropic line with the absence of the fine structure. According to the research performed by other authors for similar systems, the concentration of so-called dark centres of most of the samples was about $10^{12} - 10^{14}$ cm$^{-3}$. The EPR spectral analysis of As-Se-Sb glasses shows that as the temperature decreases from 300 to 77 K, the g-factor increases from 2.15 to 2.65, while the line width almost stays unchanged (about 954 Gs at 300 K and about 1069 Gs at 77 K). At the same temperature the intensity of the EPR spectrum of $(\text{As}_2\text{Se}_3)_{0.3}(\text{Sb}_2\text{Se}_3)_{0.5}$ is higher than the intensity of $(\text{As}_2\text{Se}_3)_{0.78}(\text{Sb}_2\text{Se}_3)_{0.22}$, which indicates a greater number of paramagnetic centres in the first sample. On the other hand, for the same compounds the line width increases inversely proportional to the content of arsenic and for $(\text{As}_2\text{Se}_3)_{0.5}(\text{Sb}_2\text{Se}_3)_{0.5}$ the line width is less than that for $(\text{As}_2\text{Se}_3)_{0.78}(\text{Sb}_2\text{Se}_3)_{0.22}$. It can be seen that with the increase in arsenic content, for the $(\text{As}_2\text{Se}_3)_{0.5}(\text{Sb}_2\text{Se}_3)_{0.5}$ sample, where the levels of arsenic are lower compared to other samples and the concentration of paramagnetic centres is higher, the line is broadened. For $(\text{As}_2\text{Se}_3)_{0.75}$
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(Sb₂Se₃)₀.₂₅ and (As₂Se₃)₀.₇₈(Sb₂Se₃)₀.₂₂ samples an additional line, with g = 2.05 and line width ∆B = 100 Gs, is observed in the EPR spectra. As it follows from the earlier work [21], one can assume that this line matches the paramagnetic centre of selenium. The spectrum of the (As₂Se₃)₀.₇₅(Sb₂Se₃)₀.₂₅ glass exhibits a broad line of irregular shape with width of 1,016 Gs, for which g = 2.20 at T = 300 K. The increase in the line width with increasing levels of arsenic in the samples indicates a structural transformation of the glass.

Using the EPR spectra one can observe the effect of “ageing” for CGS of As-Se-Sb. Figure 7 illustrates the evolution of the EPR spectra of As-Se-Sb CGS over time (7 months). The effect of “ageing” of the CGS of As-Se-Sb is manifested in the broadening of the spectral lines of the EPR, increasing their intensity, and in the appearance of additional lines in the (As₂Se₃)₀.₇₈(Sb₂Se₃)₀.₂₂ sample. The appearance of the narrow line is apparently caused by partial crystallization of the sample with time. This is confirmed by an experiment with the rotation of the ampoule with a sample in the cavity of the spectrometer, in which the shape of the EPR changes slightly.

5. Conclusion

The study of the ⁷⁵As, ¹²¹Sb and ¹²³Sb NQR and ESR spectra of chalcogenide glassy AsₓSbᵧSe₁₋ₓ₋ᵧ and GeₓSbᵧSe₁₋ₓ₋ᵧ compounds, allows one to establish the connection between the NQR frequencies and the line widths for ⁷⁵As, ¹²¹Sb and ¹²³Sb nuclei with a local structural order of CGS, to make an assumption about the presence of clusters forming in the structure of the studied glasses, to measure the EPR spectral parameters depending on the composition, on the average coordination number and on the temperature.

It is found that in the glasses of As-Se-Sb-Se system the value of the electric field gradient at the resonating nuclei grows with increasing concentration of clusters As₂Se₃, thereby increasing the NQR resonance frequencies. The large width of the NQR spectrum of As-Se-Sb-Se system is caused by the scatter of the electric field gradient caused by the dispersion of bond angles in the unit cells of As₂Se₃ and of Sb₂Se₃ in the glassy state. The observed effect of “ageing” which is manifested in the appearance of a narrow line in the EPR spectrum of As-Se-Sb-Se system is due to partial crystallization of the sample in process of time.

The ⁷⁵As NQR frequencies of both lines increase due to the growth of the average coordination number in a disordered system of Ge-As-Se: it indicates an increase of electric field gradient at the nuclei. The integrated intensity of the NQR lines for both non-equivalent positions of arsenic in the Ge-As-Se glasses and their widths are approximately equal for the average coordination number ̅r = 2.45. This fact indirectly confirms the previous hypothesis about structural transition in Ge-As-Se glass at a value of the average coordination number ̅r = 2.45 from
two-dimensional to three-dimensional structure. The appearance of narrow lines in the EPR spectrum of this glass also points to this.

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