

## AMORPHOUS, VITREOUS, AND ORGANIC SEMICONDUCTORS

# Tin Impurity Centers in Glassy Germanium Chalcogenides

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**Abstract**—Tin atoms produced by radioactive decay of  $^{119m}\text{Sn}$  and  $^{119}\text{Sn}$  impurity atoms in the structure of  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$  glasses are stabilized in the form of  $\text{Sn}^{2+}$  and  $\text{Sn}^{4+}$  ions and correspond to ionized states of the amphoteric two-electron center with negative correlation energy ( $\text{Sn}^{2+}$  is an ionized acceptor, and  $\text{Sn}^{4+}$  is an ionized donor), whereas the neutral state of the  $\text{Sn}^{3+}$  center appears to be unstable.  $^{119}\text{Sn}$  atoms produced by radioactive decay of  $^{119m}\text{Te}$  impurity atoms in the structure of  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$  glasses are stabilized at both chalcogen sites (they are electrically inactive) and germanium sites.

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## 1. INTRODUCTION

To describe the behavior of impurity atoms in chalcogenide glassy semiconductors (CGS), the concepts of the existence of two-electron centers with negative correlation energy ( $U^-$ -centers) in the glass structural network are widely used [1]. The hypothesis that such centers exist in the unordered CGS structural network makes it possible to explain the electrical, thermal, and magnetic properties of these materials (the absence of impurity conductivity, chemical potential stabilization near the midgap, and the absence of an electron paramagnetic resonance signal). Such centers were detected for the first time by the method of absorption  $^{119}\text{Sn}$  Mössbauer spectroscopy in chalcogenide glasses produced by conventional synthesis from melt, during which tin atoms enter the unordered glass network with the formation of intrinsic structural units [2]. Nevertheless, despite the large number of published studies of  $U^-$ -centers by Mössbauer spectroscopy, a basic problem remains unresolved: despite the efficiency of the theory of  $U^-$ -centers for explaining CGS electron-optical properties, such centers were not detected by Mössbauer spectroscopy directly at CGS structural network sites [3, 4]. In this respect, the study of the state of tin impurity atoms in lead chalcogenides by  $^{119m}\text{Sn}$  ( $^{119m}\text{Sn}$ ),  $^{119}\text{Sb}$  ( $^{119m}\text{Sb}$ ), and  $^{119m}\text{Te}$  ( $^{119m}\text{Te}$ ) emission Mössbauer spectroscopy is of particular interest; e.g., depending on the chemical nature of the parent atom (tin, antimony, tellurium), it became possible to introduce a daughter atom (tin) into various sublattices of lead chalcogenides [5]. By analogy, the use of emission spectroscopy with indicated isotopes allows introduction of tin atoms into

anionic and cationic structural networks of glassy germanium chalcogenides.

All these factors motivated us to study tin  $U^-$ -centers in glassy chalcogenides by  $^{119m}\text{Sn}$  ( $^{119m}\text{Sn}$ ),  $^{119}\text{Sb}$  ( $^{119m}\text{Sb}$ ), and  $^{119m}\text{Te}$  ( $^{119m}\text{Te}$ ) emission Mössbauer spectroscopy, when a radioactive parent isotope is introduced into a material under study; after its decay, the  $^{119m}\text{Sn}$  daughter atom is produced. The emission version of spectroscopy makes it possible to study impurity atoms with extremely low concentrations, which is of fundamental importance to understand the nature of tin electrical activity in lead and germanium chalcogenides.

## 2. EXPERIMENTAL

Glassy  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$  and  $\text{Ge}_{0.283}\text{Sn}_{0.002}\text{Pb}_{0.150}\text{S}_{0.565}$  and  $\text{Ge}_{0.268}\text{Sn}_{0.002}\text{Pb}_{0.170}\text{Se}_{0.560}$  alloys were synthesized at a temperature of 950°C. Melts were quenched in air. The glassy state criteria were the conchoidal fracture, the absence of lines in powder patterns, and the absence of inclusions and inhomogeneities on polished surfaces observed by an MIM-7 metallographic microscope and an MIK-1 infrared microscope. The glass composition was determined by X-ray fluorescence analysis.

Mössbauer sources were prepared by alloying ready samples with metal tin labeled by the  $^{119m}\text{Sn}$  isotope and carrier-free  $^{119}\text{Sb}$  and  $^{119m}\text{Te}$  preparations, so that the estimated concentration of impurity tin atoms in samples did not exceed  $10^{18} \text{ cm}^{-3}$ , concentrations of antimony and tellurium did not exceed  $10^{17} \text{ cm}^{-3}$ . Isotopes  $^{119m}\text{Sn}$ ,  $^{119m}\text{Te}$ , and  $^{119}\text{Sb}$  were produced by reactions  $^{118}\text{Sn}(n, \gamma)^{119m}\text{Sn}$ ,  $^{116}\text{Sn}(\alpha, n)^{119}\text{Te}$ , and

$^{119}\text{Sn}(p, n)^{119}\text{Sb}$ . Carrier-free  $^{119}\text{Sb}$  and  $^{119m}\text{Te}$  isotopes were separated by the anion exchange method.

Emission Mössbauer spectra of  $^{119m}\text{Sn}(^{119m}\text{Sn})$  were measured at 80 and 295 K using a  $\text{CaSnO}_3$  absorber (the surface density by tin is  $5 \text{ mg cm}^{-2}$ ). The spectrum with such an absorber and  $\text{Ca}^{119m}\text{SnO}_3$  source consisted of a single line with full width at half-maximum  $\Gamma = (0.79 \pm 0.01) \text{ mm/s}$ , which was taken as an instrumental spectral line width.

### 3. EXPERIMENTAL RESULTS AND DISCUSSION

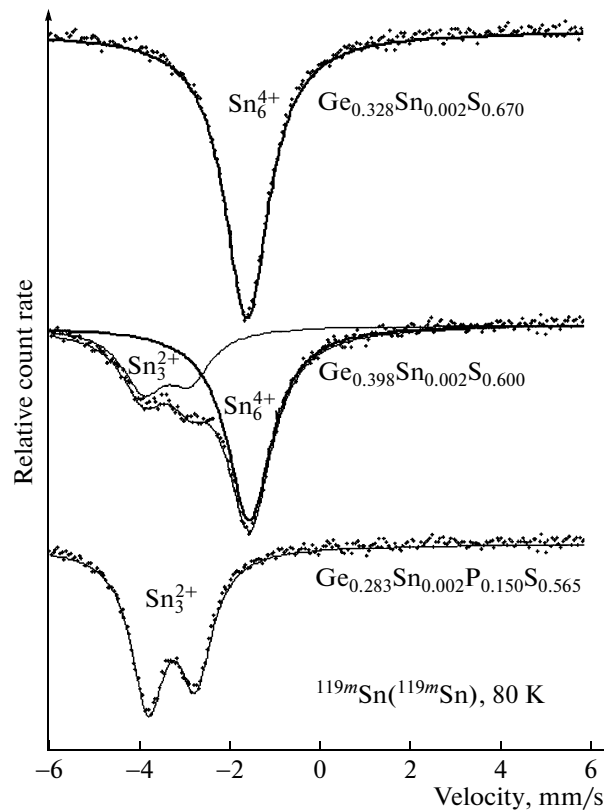
Two-electron centers with negative correlation energy can exist in three charge states:  $D^{2+}$ ,  $D^+$ , and  $D^0$  for the donor centers and  $A^+$ ,  $A^0$ , and  $A^-$  for amphoteric centers; they correspond to zero, one, and two electrons at the center, respectively. It is obvious that the intermediate charge state of such centers is unstable; for it, the disproportionation reaction occurs:  $2D^+ \rightarrow D^0 + D^{2+}$  or  $2A^0 \rightarrow A^- + A^+$ .

Therefore, identification of  $U^-$ -centers by Mössbauer spectroscopy amounts to the following: the detection of two charge states of the center, differing by  $2e$  ( $e$  is the elementary charge), and the detection of the dependence of populations of these states on the chemical potential position, and fixing the absence of an intermediate charge state of the center. If two charge states of the center have identical local structures (identical coordination numbers, identical symmetry of local environment, and identical atoms in the local environment), the  $U^-$ -center is either a two-electron donor or a two-electron acceptor (neutral and doubly ionized states are stable; singly ionized state of the center is unstable). If two charge states of the center have different local structures, the  $U^-$ -center is a two-electron amphoteric center (stable are singly ionized donor and acceptor states, the neutral state of the amphoteric center is unstable).

#### 3.1. Parent $^{119m}\text{Sn}(^{119m}\text{Sn})$ Atoms

It was assumed that impurity tin atoms isovalently substitute for germanium atoms in the glass structural network and the local tin structure reflects the local structure of germanium atoms. The emission  $^{119m}\text{Sn}(^{119m}\text{Sn})$  Mössbauer spectra of  $\text{Ge}_{0.328}\text{Sn}_{0.002}\text{S}_{0.670}$  and  $\text{Ge}_{0.328}\text{Sn}_{0.002}\text{Se}_{0.670}$  glasses consist of broadened lines; the spectral parameters of glasses are typical of spectra of tetravalent six-coordinated tin  $\text{Sn}^{4+}$ , the local environment of which contains only chalcogen atoms (such as spectra of  $\text{SnSe}_2$  and  $\text{SnS}_2$  compounds (see Figs. 1 and 2 and table).

The  $^{119m}\text{Sn}(^{119m}\text{Sn})$  emission Mössbauer spectra of  $\text{Ge}_{0.398}\text{Sn}_{0.002}\text{S}_{0.600}$  and  $\text{Ge}_{0.398}\text{Sn}_{0.002}\text{Se}_{0.600}$  glasses are superpositions of the broadened spectral line (corre-

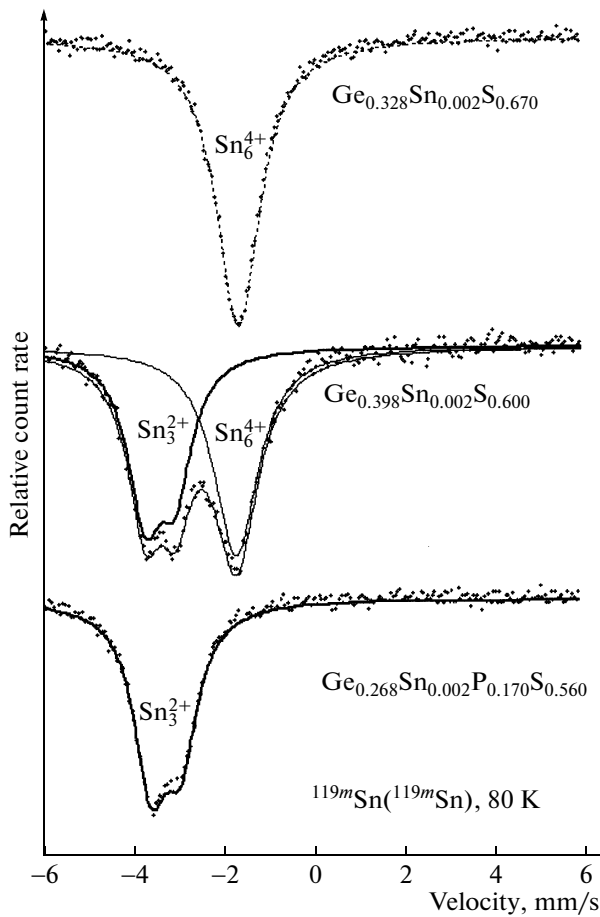


**Fig. 1.** Emission  $^{119m}\text{Sn}(^{119m}\text{Sn})$  Mössbauer spectra of  $\text{Ge}_{0.328}\text{Sn}_{0.002}\text{S}_{0.670}$ ,  $\text{Ge}_{0.398}\text{Sn}_{0.002}\text{S}_{0.600}$ , and  $\text{Ge}_{0.283}\text{Sn}_{0.002}\text{Pb}_{0.150}\text{S}_{0.565}$  glasses. The spectrum decomposition into the components corresponding to  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  is shown. The melt was quenched from  $900^\circ\text{C}$  in air.

sponding to  $\text{Sn}_6^{4+}$  centers) and the quadrupole doublet corresponding to tin(II), the parameters of which are typical of the spectra of compounds of divalent three-coordinated tin  $\text{Sn}_3^{2+}$  the local environment of which contains only chalcogen atoms (such as spectra of  $\text{SnSe}$  and  $\text{SnS}$  compounds) (see Figs. 1 and 2 and table).

Finally, the  $^{119m}\text{Sn}(^{119m}\text{Sn})$  emission Mössbauer spectra of glasses containing lead ( $\text{Ge}_{0.283}\text{Sn}_{0.002}\text{Pb}_{0.150}\text{S}_{0.565}$  and  $\text{Ge}_{0.268}\text{Sn}_{0.002}\text{Pb}_{0.170}\text{Se}_{0.560}$ ) are quadrupole doublets corresponding to  $\text{Sn}_3^{2+}$  centers (Figs. 1 and 2).

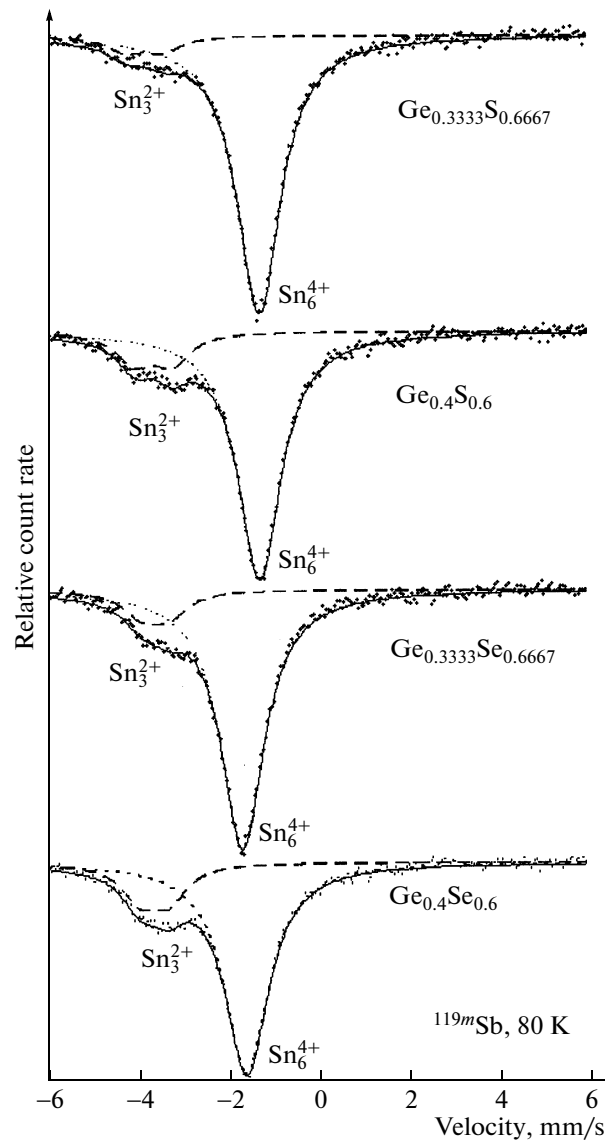
Thus, the valence and coordination states of tin atoms produced by radioactive decay of  $^{119m}\text{Sn}$  in  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$  glasses depend on both the chalcogen content in glass (in glasses enriched with chalcogen, tin stabilizes only in the  $\text{Sn}_6^{4+}$  state; in glasses depleted of chalcogen, tin is stabilized in  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  states) and the lead content (in these glasses,



**Fig. 2.** Emission  $^{119m}\text{Sn}(^{119m}\text{Sn})$  Mössbauer spectra of  $\text{Ge}_{0.328}\text{Sn}_{0.002}\text{S}_{0.670}$ ,  $\text{Ge}_{0.398}\text{Sn}_{0.002}\text{S}_{0.600}$ , and  $\text{Ge}_{0.268}\text{Sn}_{0.002}\text{Pb}_{0.170}\text{S}_{0.560}$  glasses. The spectrum decomposition into the components corresponding to  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  is shown. The melt was quenched from  $900^\circ\text{C}$  in air.

tin stabilizes only in the  $\text{Sn}_3^{2+}$  state). The appearance of tin(III) was not observed in Mössbauer spectra of glasses. Such behavior of tin impurity atoms can be explained by assuming that tin in the glass structure forms  $U^-$ -centers of amphoteric type:  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  states correspond to the ionized donor and acceptor centers of tin, respectively, while the  $\text{Sn}^{3+}$  neutral center state appears unstable.

The effect of the glass composition on the ratio of  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  center concentrations can be explained within the model according to which excess chalcogen atoms (with respect to stoichiometric  $\text{GeS}$  and  $\text{GeSe}$  compositions) in the  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$  glass structure are shallow acceptors, while lead atoms are shallow donors. Then an increase in the chalcogen concentration should result in an increase in the frac-



**Fig. 3.** Mössbauer spectra  $\text{Ge}_{0.4}\text{S}_{0.6}^{119}\text{Sb}$ ,  $\text{Ge}_{0.3333}\text{S}_{0.6667}^{119}\text{Sb}$ ,  $\text{Ge}_{0.4}\text{Se}_{0.6}^{119}\text{Sb}$ , and  $\text{Ge}_{0.3333}\text{Se}_{0.6667}^{119}\text{Sb}$ . Positions of lines corresponding to  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  centers are shown. The melt was quenched from  $900^\circ\text{C}$  in air.

tion of ionized donor centers of  $\text{Sn}_6^{4+}$  tin and lead introduction into the glass composition should lead to tin stabilization in the  $\text{Sn}_3^{2+}$  state. Exactly this effect is observed experimentally.

### 3.2. Parent $^{119}\text{Sb}$ Atoms

The  $^{119}\text{Sb}(^{119m}\text{Sn})$  Mössbauer spectra of  $\text{Ge}_{0.3333}\text{S}_{0.6667}$ ,  $\text{Ge}_{0.4}\text{S}_{0.6}$ ,  $\text{Ge}_{0.3333}\text{Se}_{0.6667}$ , and  $\text{Ge}_{0.4}\text{Se}_{0.6}$  glasses (Fig. 3) are the superposition of a broadened

Parameters of emission ( $^{119m}\text{Sn}$ ( $^{119m}\text{Sn}$ ),  $^{119}\text{Sb}$ ( $^{119m}\text{Sn}$ ),  $^{119m}\text{Te}$ ( $^{119m}\text{Sn}$ )) and absorption ( $^{119}\text{Sn}$ ) Mössbauer spectra of glassy alloys and crystalline compounds of tin at 80 K

Composition	Parent isotope	$\text{Sn}_6^{4+}$ centers			$\text{Sn}_3^{2+}$ centers				$\text{Sn}^0$ centers		
		IS, mm/s	$G$ , mm/s	$S$ , rel. units	IS, mm/s	QS, mm/s	$G$ , mm/s	$S$ , rel. units	IS, mm/s	$G$ , mm/s	$S$ , rel. units
$\text{Ge}_{0.328}\text{Sn}_{0.002}\text{S}_{0.670}$	$^{119m}\text{Sn}$	1.56	1.21	1.00							
$\text{Ge}_{0.398}\text{Sn}_{0.002}\text{S}_{0.600}$	$^{119m}\text{Sn}$	1.57	1.22	0.65	3.42	1.02	0.90	0.35			
$\text{Ge}_{0.283}\text{Sn}_{0.002}\text{Pb}_{0.150}\text{S}_{0.565}$	$^{119m}\text{Sn}$				3.29	1.06	0.90	1.00			
$\text{Ge}_{0.328}\text{Sn}_{0.002}\text{Se}_{0.670}$	$^{119m}\text{Sn}$	1.73	1.17	1.00							
$\text{Ge}_{0.398}\text{Sn}_{0.002}\text{Se}_{0.600}$	$^{119m}\text{Sn}$	1.75	1.19	0.43	3.44	0.68	0.90	0.57			
$\text{Ge}_{0.268}\text{Sn}_{0.002}\text{Pb}_{0.170}\text{Se}_{0.560}$	$^{119m}\text{Sn}$				3.30	0.67	0.90	1.00			
$\text{Ge}_{0.33}\text{S}_{0.67}$	$^{119}\text{Sb}$	1.36	1.17	1.00							
$\text{Ge}_{0.4}\text{S}_{0.6}$	$^{119}\text{Sb}$	1.35	1.15	0.81	3.71	0.81	0.96	0.19			
$\text{Ge}_{0.33}\text{Se}_{0.67}$	$^{119}\text{Sb}$	1.67	1.16	1.00							
$\text{Ge}_{0.4}\text{Se}_{0.6}$	$^{119}\text{Sb}$	1.65	1.19	0.76	3.71	0.61	0.97	0.24			
$\text{Ge}_{0.33}\text{S}_{0.67}$	$^{119m}\text{Te}$	1.36	1.38	0.20					2.47	1.35	0.80
$\text{Ge}_{0.4}\text{S}_{0.6}$	$^{119m}\text{Te}$	1.36	1.39	0.18					2.45	1.34	0.82
$\text{Ge}_{0.33}\text{Se}_{0.67}$	$^{119m}\text{Te}$	1.66	1.33	0.16					2.51	1.32	0.84
$\text{Ge}_{0.4}\text{Se}_{0.6}$	$^{119m}\text{Te}$	1.67	1.32	0.17					2.49	1.34	0.83
SnS	$^{119}\text{Sn}$				3.44	0.90	0.80	1.00			
SnSe	$^{119}\text{Sn}$				3.45	0.65	0.80	1.00			
SnS <sub>2</sub>	$^{119}\text{Sn}$	1.30	0.80	1.00		0.40					
SnSe <sub>2</sub>	$^{119}\text{Sn}$	1.65	0.80	1.00		≤0.30					
SnAs	$^{119}\text{Sn}$	2.70	0.80			≤0.20			2.70	0.80	1.00
Errors		±0.01	±0.02	±0.02	±0.01	±0.02	±0.02	±0.02	±0.01	±0.02	±0.02

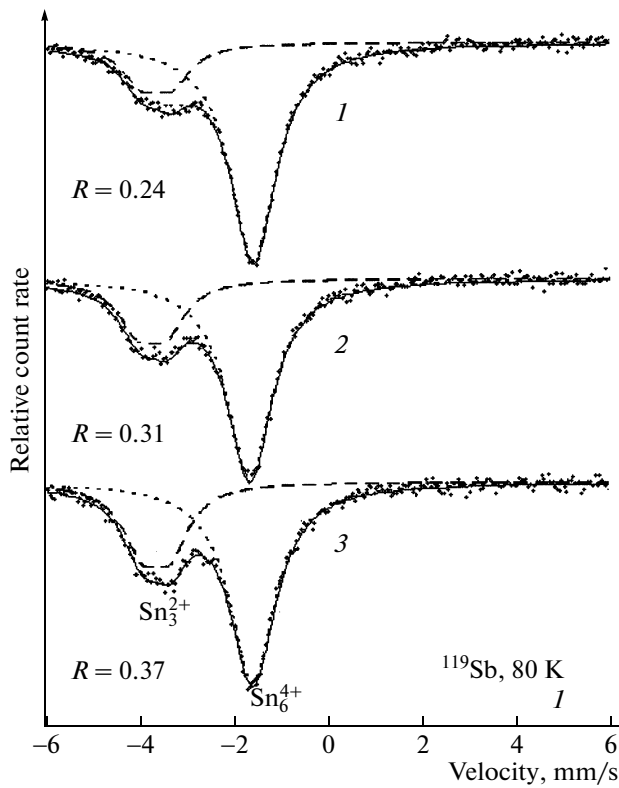
Note: IS is the isomer shift, QS is quadrupole splitting,  $G$  is the spectral linewidth, and  $S$  is the area under the spectrum.

line the parameters of which correspond to  $\text{Sn}_6^{4+}$  and a quadrupole doublet of which the parameters correspond to  $\text{Sn}_3^{2+}$ . Populations of  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  states depend on quenching conditions: the fraction of  $\text{Sn}_3^{2+}$  centers increases with the melt temperature and quenching rate (see Fig. 4 and table). It can be concluded that tin atoms produced by  $^{119}\text{Sb}$  radioactive decay in the  $\text{Ge}_{0.3333}\text{S}_{0.6667}$ ,  $\text{Ge}_{0.4}\text{S}_{0.6}$ ,  $\text{Ge}_{0.3333}\text{Se}_{0.6667}$ , and  $\text{Ge}_{0.4}\text{Se}_{0.6}$  glass structure form amphoteric  $U^-$ -centers, where  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  states correspond to singly ionized donor and acceptor, respectively. The electri-

cal activity of tin centers is confirmed by the dependence of the fine structure of  $^{119}\text{Sb}$ ( $^{119m}\text{Sn}$ ) spectra on quenching conditions: the density of the donor-type point defect and  $\text{Sn}_3^{2+}$  state population increase with the melt temperature and quenching rate.

### 3.3. Parent $^{119m}\text{Te}$ Atoms

The typical  $^{119m}\text{Te}$ ( $^{119m}\text{Sn}$ ) spectra of glassy germanium chalcogenides are shown in Fig. 5; the results of their processing are given in the table. The spectra represent the superposition of two broadened lines. The parameters of the more intense line (its area is 80% of

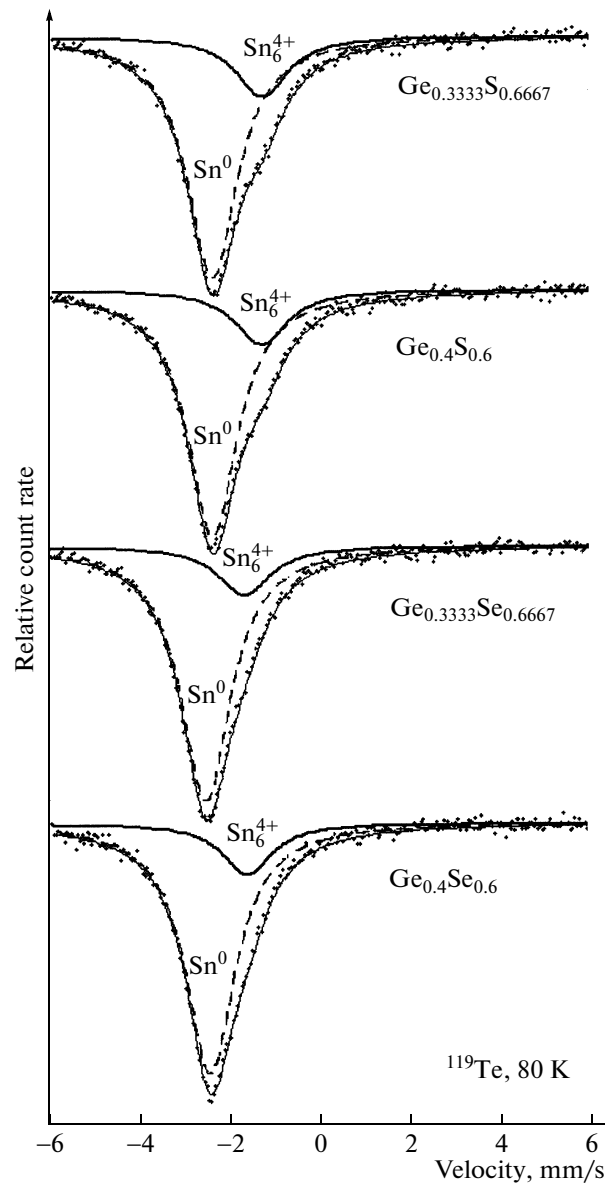


**Fig. 4.** Mössbauer spectra  $\text{Ge}_{0.4}\text{Se}_{0.6}^{119}\text{Sb}$ . Positions of lines corresponding to  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  centers are shown. The melt was quenched (1) from  $900^\circ\text{C}$  in air, (2) from  $1050^\circ\text{C}$  in air, and (3) from  $1050^\circ\text{C}$  by pouring onto a metal plate.  $R = S(2)/[S(2) + S(4)]$ , where  $S(4)$  and  $S(2)$  are the areas under the  $\text{Sn}_6^{4+}$  and  $\text{Sn}_3^{2+}$  spectra.

the entire spectrum area) are close to the parameters of spectra of intermetallic tin compounds (such as  $\text{SnAs}$ ). This spectrum should be attributed to  $\text{Sn}^0$  tin centers produced by the decay of  $^{119m}\text{Te}$  parent atoms located at chalcogen sites. The parameters of the less intense line depend on the chalcogen chemical nature (see table). This line corresponds to tin atoms displaced due to the recoil energy (the tin atom recoil energy after  $^{119m}\text{Te}$  decay is  $\sim 24$  eV) from chalcogen sites to germanium sites.

#### 4. CONCLUSIONS

Impurity tin atoms in the structure of glassy germanium chalcogenides substitute germanium atoms. The valence and coordination state of tin atoms produced by radioactive decay of  $^{119m}\text{Sn}$  atoms in  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$  glasses depends on both chalcogen and lead contents in glass. Tin(III) was not observed in Mössbauer spectra of glasses. Such behavior of impurity tin atoms is attributed to the fact that tin forms amphoteric-type  $U^-$ -centers in the  $\text{Ge}_x\text{S}_{1-x}$  and  $\text{Ge}_x\text{Se}_{1-x}$



**Fig. 5.** Mössbauer spectra  $\text{Ge}_{0.4}\text{S}_{0.6}^{119m}\text{Te}$ ,  $\text{Ge}_{0.3333}\text{S}_{0.6667}^{119m}\text{Te}$ ,  $\text{Ge}_{0.4}\text{Se}_{0.6}^{119m}\text{Te}$ , and  $\text{Ge}_{0.3333}\text{Se}_{0.6667}^{119m}\text{Te}$ . Positions of lines corresponding to  $\text{Sn}_6^{4+}$  and  $\text{Sn}^0$  are shown. The melt was quenched from  $900^\circ\text{C}$  in air.

glass structure: the  $\text{Sn}_4^{4+}$  and  $\text{Sn}_3^{2+}$  states correspond to ionized donor and acceptor centers, respectively, while the state of the  $\text{Sn}^{3+}$  neutral center appears unstable.

Impurity antimony atoms in the structure of glassy germanium chalcogenides form intrinsic structural units. Tin atoms produced by radioactive decay of  $^{119}\text{Sb}$  atoms play the role of amphoteric two-electron centers with negative correlation energy; the  $\text{Sn}_3^{2+}$  and

$\text{Sn}_6^{4+}$  states correspond to singly ionized acceptors and donors, respectively, while neutral states of these centers ( $\text{Sn}_6^{3+}$  and  $\text{Sn}_3^{3+}$ ) are found to be unstable.

Most  $^{119m}\text{Sn}$  atoms produced by radioactive decay of parent  $^{119m}\text{Te}$  atoms in glassy germanium chalcogenides are at chalcogen sites and are electrically inactive. The significant recoil energy of daughter atoms in the case of  $^{119m}\text{Te}$  decay results in the appearance of displaced  $^{119m}\text{Sn}$  atoms.

#### ACKNOWLEDGMENTS

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