

# Effect of gamma irradiation on microstructure of the layered $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$

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X-ray analysis of layered  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  crystal has been investigated before and after gamma irradiation. Single crystal  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  was irradiated with a  $^{60}\text{Co}$  gamma source which had an energy of 1.33 MeV. The dose rate was  $D = 0.35$  Gy/s and the absorption dose was from 1 kGy up to 30 kGy. The thermogravimetric analysis, differential scanning calorimetric, differential thermogravimetric and differential thermal analysis were performed on the samples. The thermodynamic analysis was evaluated for the thermally stable temperature range for unirradiated and irradiated  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound. The results showed the splitting of GeNdS sample in the manner  $\rightarrow \text{GeNd} + \text{Ge}_{1-x}\text{S}$  at the temperature range of  $644^\circ\text{C} \leq T \leq 675^\circ\text{C}$  which leads to mass reduction by 0.29%.

*Keywords:*  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound; gamma irradiation time; SEM; X-ray diffraction; lattice parameters.

## 1. Introduction

The study of functional materials with various physical properties has played an important role in modern condensed state of physics. From this point of view, materials with magnetic properties such as hexaferrites,<sup>1-4</sup> single-phase metal materials<sup>5-8</sup> and single-phase oxide materials<sup>9,10</sup> have a great importance. Germanium

monosulfide belongs to  $A^{IV}B^{VI}$  families of semiconductors with  $p$ -type conductivity and is related to the orthorhombic-type structure SnS with the space group  $Pcmm$  ( $D_{2h}^{16}$ ). GeS also has a layered crystal structure, where the atomic layers are connected only by Van der Waals forces.<sup>11</sup> Thus, electron levels on the surface of a GeS single crystal are completely unoccupied, due to the fact that the material surface is characterized by high chemical stability. Increasing interest in these materials is due to their photoelectric properties, as it has been suggested that the material could be used successfully as a photosensitive film in TV camera tubes,<sup>12,13</sup> in electrical memory devices,<sup>14</sup> as a medium for recording holograms,<sup>15</sup> etc. Owing to its small size and thin structure, GeS allows the increased capacity of lithium-ion batteries. Such material is suitable as a raw material in the production of small-sized solar super capacitors.<sup>16</sup> The single crystal GeS compound grown by chemical vapor deposition is a promising nanomaterial for devices with high sensitivity of visible light.<sup>17,18</sup> The traditional approach for expanding the field of practical application of semiconductor materials is a process based on doping them with impurities. The main task in this case is choosing the right dopant. From this point of view in contrast to other impurities, rare-earth element (REE) impurities are characterized by low limit solubility of the crystal lattice and chemical activity.<sup>19,20</sup> As a result of the Coulomb and chemical interactions with the atoms of the main substance, REE forms various kinds of complexes. Many of the complexes formed as a result of such interactions have a sufficiently high stability and have a significant effect on the properties of the semiconductor, such as the effective scattering centers for ionizing radiation. With the study of the electronic structure of REE atoms in semiconductors, it must be noted that the one remarkable property is their ability to produce "cleaning up" the material under certain conditions. In this way, it is possible to decrease the concentration of background impurities in  $A^4B^6$  compounds by 2–3 orders and significantly increase the mobility of electrons. The main difficulty of working with REE lies in their extreme chemical activity. Since REE is covered with a film of the corresponding oxide, its removal is almost impossible. Gamma irradiation is a promising technological process for the manufacture of semiconductor devices.<sup>21</sup> During irradiation of semiconductor with low dose gamma radiation, a condition whereby the structure becomes highly disorder is created. The reliability of the observed effect is confirmed by X-ray diffraction (XRD) and electron microscopic studies. The physical properties of solid materials are directly related with their crystal structures at their atomic level. Therefore, it is important to investigate the crystal structure of each material and study the occurring changes under external influences. In this work, the systematic investigation of the GeS compound doped with Nd atoms is studied, and the analysis of the samples was performed using a Scanning Electron Microscope (SEM), XRD and Thermal analysis method.

## 2. Materials and Methods

The starting material was Germanium with a specific resistance of  $50 \Omega \cdot \text{cm}$ , sulfur of the mark “B5” and neodymium of the mark “Nd-2.” The stoichiometric mixture of these elements was loaded into a long quartz tube of about 10–15 in length (OD: 1.0–2.0 cm) and evacuated to  $10^{-3}$  mm (Hg). In order to avoid an explosion, germanium was powdered, and the amount of the substance was limited to about 10–15 g. The synthesis process was carried out in two stages: in the first step, ampoule was kept in a furnace and heated at a rate of 3–5 Gy/min up to  $300^\circ\text{C}$  and kept at this temperature for 10–12 h. Then, the temperature was increased at a rate of 2–3 Gy/s until germanium melted completely. The temperature (melting point) was kept constant for 18–20 h. The temperature of the upper part of the furnace was  $50^\circ\text{C}$  higher and the bottom part  $50^\circ\text{C}$  lower than the melting point of the sample. After this process, the ampoule was lowered down at a growth with speed of 2–3 mm/h. The single crystals were grown in the form of plane-parallel plates with dimensions of  $10 \times 8 \times 0.1 \text{ mm}^3$  or with needles. Grown from large ingots, layered monocrystal were easily exfoliated immediately before the measurements along the plane perpendicular to the  $c$ -axis. Due to their good mirror surface, they had not undergone additional processes to improve the surface by further mechanical and chemical processing. X-ray powder diffraction (XRD) patterns were recorded at room temperature on a Bruker D8 Focus diffractometer with  $\text{Cu K}\alpha$  ( $\lambda = 1.5418 \text{ \AA}$ ) radiation.<sup>22,23</sup> Surface morphology of single crystals was studied with SEM using a SIGMA VP VAT electron microscope.<sup>24</sup> The thermal property was carried out in a Perkin Elmer, Simultaneous Thermal Analyzer, STA 6000. In the thermal analysis, the sample was heated from  $30^\circ\text{C}$  to  $900^\circ\text{C}$  at a heating rate of  $20 \text{ mL/min}$  with Ar gas flow rate of  $5^\circ\text{C/min}$ . After heating the sample, it was cooled in the PolyScience analyzer cooling system. Differential Thermogravimetric (DTG) and Differential Thermal Analysis (DTA) were carried out using “Pyris Thermal Analysis Manager” program. The error of weight determination did not exceed 1.5% at  $30^\circ\text{C}$  and 1% at  $900^\circ\text{C}$ .<sup>25–27</sup> Irradiation of samples with gamma quanta was carried out at room temperature on an RCDED-20000 installation using a  $^{60}\text{Co}$  source with a phase power in the irradiation zone of  $\sim 1.37 \text{ P/c}$ .<sup>28</sup>

## 3. Results and Discussion

### 3.1. X-ray diffraction

The XRD patterns of  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  of crystal structure before and after irradiation with different doses are shown in Fig. 1. The XRD pattern was analyzed with a Rietveld method (with the help of a FullProf Program). From the XRD analysis, it was determined that in ambient conditions,  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  has orthorhombic crystal structure (specific gravity  $Pnma$ ) with lattice parameters:  $a = 4.321(6) \text{ \AA}$ ,  $b = 3.652(2) \text{ \AA}$  and  $c = 10.493(7) \text{ \AA}$  ( $R_p = 9.73$ ,  $R_{wp} = 11.52$  and  $\chi^2 = 8.22\%$ ). The XRD pattern after radiation with absorption dose  $D = 30 \text{ kGy}$  in the

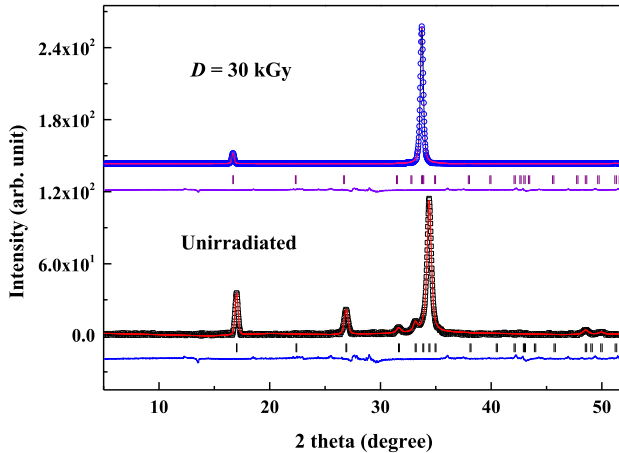


Fig. 1. X-ray diffraction spectra of  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  measured at room temperatures. Experimental points, calculated profile, difference curve and calculated positions of diffraction peaks are shown.

spectrum showed peaks correspondingly to central (201) plane  $2\theta = 26.9^\circ$ , (210) plane  $2\theta = 31.6^\circ$ , (301) plane  $2\theta = 33.2^\circ$ , (411) plane  $2\theta = 48.5^\circ$  and (302) plane  $2\theta = 49.9^\circ$ . It is known that the decrease in the number of peaks in the XRD spectrum is related to the increasing of symmetry. The obtained lattice parameters of crystal structure with  $Pnma$  spatial group which crystallized in orthorhombic symmetry using Fullprof programm were  $a = 4.319(3) \text{ \AA}$ ,  $b = 3.651(4) \text{ \AA}$  and  $c = 10.492(5)$  ( $R_p = 10.21$ ,  $R_{wp} = 11.02$  and  $\chi^2 = 9.16\%$ ). During the doping of GeS compound with Nd atoms, a number of additional effects associated with defect formation occurred due to the deviation of the composition from the stoichiometric. Noticeable amounts of impurities were introduced by replacing Ge atoms which lead to an additional change in the ratio of the concentration of the main components in the crystal. Because of the significant difference in the sizes of the impurity atoms ( $r_{\text{Nd}} = 0.96 \text{ \AA}$ ,  $r_{\text{Ge}} = 0.72 \text{ \AA}$ ), the doping process may have generated additional defects (own point defects) and a change of their distribution shape in the crystal.<sup>29</sup> Also, a large number of cationic vacancies ( $10^{17}\text{--}10^{18} \text{ cm}^{-3}$ ) can affect the character of the impurity atoms into the GeS lattice. In the range of low concentrations, Nd atoms preferentially dissolve in vacancies. Their solubility in vacancies depends on the concentration of vacancies in the initial material. It must be noted that the solubility of impurities in vacancies is limited and always less than the concentration of vacancies. Also, in the presence of free vacancies, it is possible to dissolve Nd impurities in significant quantities by replacing germanium in the lattice nodes. Taking into account these data, we can speculate that the main mechanism of dissolution of low concentrations of Nd impurity in germanium monosulfide leads to the “healing” of cationic vacancies. When high-energy charged particle passes through a crystal, the main mechanisms of inhibition are

elastic collisions with nuclei and inelastic electrons. The electronic mechanism was dominating during gamma irradiation.<sup>30</sup> Thus, at a sufficiently high energy of the incident particle, the target atom is displaced from the angular position, which leads to the appearance of an interstitial atom and a vacancy (Frenkel pair). There was an accepted point of view according to which physical properties of gamma irradiated samples with smaller orders of charge carriers concentration (of particles) do not cause any changes.<sup>31,32</sup> However, the experiment result reported in Ref. 33 indicated that the process of interaction of ionizing radiation with crystals does not correspond to the accepted concepts in the case when the absorbed dose is  $\sim 105$  Gy. It turns out that irradiation with gamma rays (as indicated by the absorbed dose of ionizing radiation of semiconductor crystals) does not lead to the accumulation of defects, on contrary, it leads to their elimination and regulation of crystal structure of materials.<sup>34</sup> The occurrence of restructuring in the crystal structure during low dose irradiation is due to the release by accumulated energy in the crystal. A decrease in the number of defects in a crystal during irradiation is accompanied by thermal separation which occurs due to the annihilation and restructuring of defects.<sup>35</sup> After irradiation, structural change in GeS compound doped with Nd atoms was observed. Since after low dose irradiation (30 kGy) as can be seen from Fig. 1, several peaks disappear. It is known that during investigation, the amount of oxygen atoms in the composition of compounds has the ability to change.<sup>36,37</sup> By XRD analysis, oxidation on the surface of the  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound was not observed. Such physical processes occur mainly in the investigation of lattice parameters in the high-temperature range.<sup>38</sup> For more detailed studying of crystal structure, atomic coordinates are needed to carry out the high-intensity neutron diffraction investigation.<sup>39</sup> Because, the vacancy occupying of atoms and interatomic positions affects not only on the crystal structure, but also on the crystallite dimensions.<sup>40</sup>

### 3.2. *Surface analysis*

In addition to understanding how such impurities affect the crystal surface structure of GeS, it is very essential to study the surface morphology of a sample. In the crystal, the combination of small associations of point defects appears in a form of blocks of light spots. Under the action of very low doses, the size of crystal blocks increases (Fig. 2). Qualitative and quantitative analyses of the processes occurring in a  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  crystal during gamma radiation in low doses (30 kGy) can be explained as follows. As a result of gamma irradiation, gamma quanta create electron-hole pairs in a crystal. These pairs in turn exist for quite a long time in semiconductor crystals by migrating over a crystal. They are captured by the defects and they form charged interstitial atoms and vacancies or clusters. In this way, charged defects intensively interacted with each other. During the interaction processes, the defects of the same name merged to form larger complexes of interstitial atoms or vacancies. While, in the case of defects with different names, annihilation

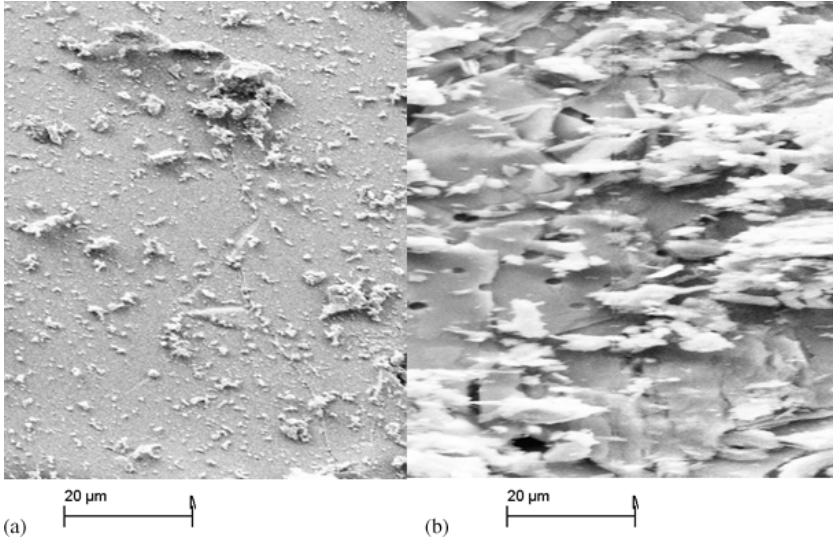


Fig. 2. Surface of single crystal  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  at SEM microscope: (a) unirradiated and (b)  $D = 30$  kGy.

occurs. Additionally, the occurrence of annihilation of electron and hole leads to the generation of photon, which is able to interact with the complexes (which include REE and oxygen atoms) and break them up. Thus, the released oxygen leaves the crystal, and the appearance of free interstitial atom due to the annihilation with vacancies created the so-called Frenkel pair. During the annihilation of Frenkel pair, the released energy forms new electron–hole pairs. These processes continue with the capture of the latter by the annihilated defects.<sup>41</sup> The described process changes the state of the crystal and leads to the ordering of the crystal structure. As can be seen from the value of the cell parameters, low dose gamma irradiation has negligible effect on the parameters of the unit cell. Thus, under the action of low dose gamma radiation, an ordered state of the  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  crystal is formed, where neodymium atoms are also located in the crystal nodes.

### 3.3. Thermal analysis

Spectral characterization and study of thermal behavior kinetics of  $\text{Ge}_{0.995}\text{Nd}_{0.005}$  compound by thermogravimetric (TG) analysis, Differential Scanning Calorimetric (DSC), DTG and DTA spectra are shown in Fig. 3. It is known that the change in the dynamics of DSC, DTG and DTA spectra are dependent on the kinetics of TG spectra. Although in the range of  $T = 50\text{--}750^\circ\text{C}$ , the kinetics of TG spectra of  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  combination are in one phase, but spectra consist of three successive thermal stability areas. Furthermore, thermodynamic analysis was evaluated to be thermally stable at the temperature range of  $50^\circ\text{C} \leq T \leq 570^\circ\text{C}$  for  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$ . However, the mechanism of the end effect with decomposition

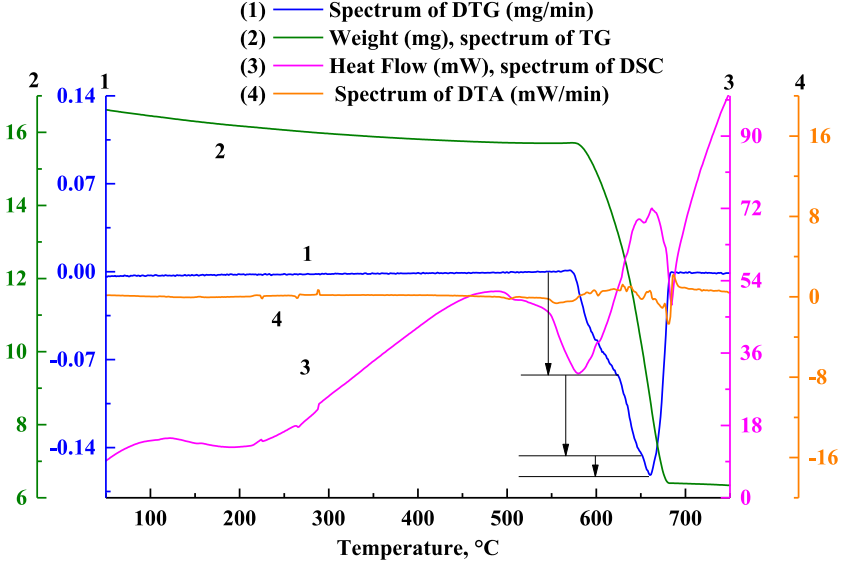


Fig. 3. (Color online) TG, DTG, DSC and DTA spectra of  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  at normal condition.

character began at  $T = 570^\circ\text{C}$  and completed at  $T = 570^\circ\text{C}$  with central peak  $T = 665^\circ\text{C}$ . It consists of three parts shown with small arrows in Fig. 3. Physical parameters such as energy of field 6582 mJ, enthalpy 419.23 J/g and the activation energy 0.42 kJ/mol were obtained. It is known that GeS compound has splitting which occurs in this manner:  $\text{GeS} \rightarrow \text{Ge} + \text{Ge}_{1-x}\text{S}$  at the central peak is situated at  $T = 665^\circ\text{C}$ .<sup>42</sup> As reported in Ref. 42, experiment work in the manner  $\text{GeS} \rightarrow \text{Ge} + \text{Ge}_{1-x}\text{S}$ , in which 60% of  $\text{Ge}_{1-x}\text{S}$  was splitting. We would like to mention that in our experimental investigations, this value is about 59.2% which is in good agreement with the previous<sup>42</sup> experimental work. TG, DSC, DTG and DTA spectra of crystal structure  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  depend on the temperature during irradiation with the  $^{60}\text{Co}$  gamma source using a dose rate of  $D = 0.35$  Gy/s, with energy line 1.17 and 1.33 MeV and 30 kGy absorption dose as shown in Fig. 4. In contrast to the unirradiated sample, the mass kinetics of the  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound are thermally stable at a temperature range of  $50^\circ\text{C} \leq T \leq 500^\circ\text{C}$ . However, the observed exoeffect in the range of  $500\text{--}644^\circ\text{C}$  is characterized by oxidation reaction obtained in DSC and TDG spectra. During the oxidation reaction, the mass of the sample is increased by 4.52%. This increase is related to the oxygen atoms which partake in the reaction and their quantity is about  $5.5 \cdot 10^{20}$ . Also, the thermophysical parameters of this effect are the following: the energy of the field 5613 mJ, the enthalpy 247.18 J/g and activation energy 0.25 kJ/mol. The observed splitting of GeNdS sample following this order:  $\text{GeNdS} \rightarrow \text{GeNd} + \text{Ge}_{1-x}\text{S}$  at the temperature range of  $644^\circ\text{C} \leq T \leq 675^\circ\text{C}$  is the result of the reduced mass by 0.29%.

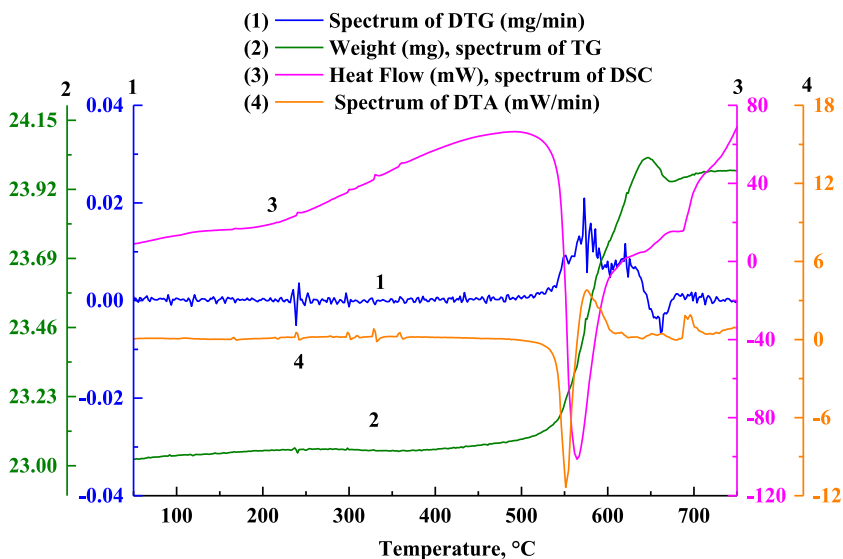


Fig. 4. (Color online) TG, DTG, DSC and DTA spectra of  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  at  $D = 30$  kGy radiation.

#### 4. Conclusions

The effect of gamma rays on surface morphology, crystalline structure and thermal properties of  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound has been studied. Investigation showed that irradiation with absorption dose of 30 kGy leads to the occurrence defects on the surface of the  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound. Furthermore, decreasing in lattice parameters was observed due to the irradiation. The observed decrement in parameter  $a$  by 0.05%, in  $b$  by 0.02% and in  $c$  by 0.001% indicates that reduction took place mainly in **ab** plane. As a result of the thermal analysis, effect of gamma rays did not indicate any significant changes in the DTA spectrum of the  $\text{Ge}_{0.995}\text{Nd}_{0.005}\text{S}$  compound. However, significant changes have been observed in TG analysis, which was associated with the influence of gamma rays on the change mechanism of mass.

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