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## AFM-MICROSCOPY STUDY OF THE SURFACE OF GAMMA-IRRADIATED GaS AND GaS:Yb LAYERED CRYSTALS

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Abstract: For the first time, information on the surface relief of undoped GaS and doped GaS:Yb single crystals subjected to gamma irradiation was obtained using atomic force microscopy (AFM). It was found that GaS is characterized by a non-uniform distribution of irregularities with different heights and periodicities, and when doping crystals with Yb atoms, the distribution of irregularities becomes more orderly, the height and periodicity of irregularities decreases. Irradiation with gamma-quanta of GaS at doses of  $\Phi_{\gamma} < 140$ krad and doping with Yb atoms lead to rearrangement of defects, resulting in a decrease in a single free volume of irregularities, and in doses above  $\Phi_{\gamma} > 140$  krad increase single crystals are the most radiation-resistant.

*Keywords:* layered GaS and GaS:Yb single crystals, relief,  $\gamma$ -irradiation, surface, defect, AFM microscopy

#### 1. Introduction

Layered  $A^3B^6$  semiconductors, in particular, gallium sulfide single crystals (GaS) are promising materials for radiation detectors of various types. Based on these single crystals, radiation detectors of gamma quanta operating at room temperature [1] are fabricated. The increased interest in these compounds is due to the anisotropy of their crystalline structure, which allows obtaining perfect faces with a sufficiently low density of surface states, which is important for obtaining high-quality heterojunctions.

One of the effective methods for modifying the surface of layered gallium sulfide single crystals is to irradiate it with  $\gamma$ -quanta [2-6]. The depth of penetration of gamma-quanta is comparable to the value of the inverse light absorption coefficient (~ 10<sup>2</sup> nm), which leads to the desorption of gases from the surface and recharging the surface-active centers. This factor is decisive in many processes occurring near the surface of the crystal. Therefore, studying the effect of external influences, including gamma radiation, on the edge photoconductivity (EP) of a defective semiconductor, one can establish the role of surface heterogeneity and roughness during its formation [4]. The most informative method for studying the surface of semiconductors is the method of atomic force microscopy (AFM) [7,8].

Based on these considerations, the present work presents the results of the microscopic (AFM) study of changes in the surface relief of gamma-irradiated layered GaS and GaS:Yb single crystals.

## 2. Experimental technique

Single crystals of p-GaS were grown by the method of directional solidification of the melt (the vertical version of the Bridgman method). When growing GaS, an excess of sulfur (1.5%) was used to determine the possibility of filling vacancies with sulfur atoms. The

resistivity of the samples obtained along with and perpendicular to the C axis at room temperature was  $3 \times 10^7$  and  $2 \times 10^9$  Om·sm, respectively. The doping of Yb was carried out in the process of crystal growth, and the concentration of Yb in crystals was  $N_{Yb} \sim 10^{18}$  sm<sup>-3</sup>. Indium, which was smelted on the surface of gallium sulfide at a temperature of 150°C, was used as the ohmic contact.

Microstructural and X-ray phase analyzes showed that the obtained crystals were homogeneous and did not contain crystalline inclusions [2, 3].

Microscopic studies of the surface relief of the initial gamma-irradiated GaS and GaS:Yb samples were carried out with an atomic force microscope (AFM). For this purpose, twodimensional (2d) and three-dimensional (3d) surface AFM images were obtained, as well as histograms (distribution curves of surface images on the size of irregularities) in the horizontal and vertical directions of the selected section ( $100 \times 100$  nm).

Samples were irradiated with  $\gamma$ -quanta from a <sup>60</sup>Co source at room temperature with a dose rate d  $\Phi_{\gamma}/dt = 15.66$  rad / s. In this case, the absorbed dose was  $\Phi_{\gamma} = 30-200$  krad. The samples were irradiated with doses of 30,50,100,140 and 200 krad [9].

#### 3. Results and its discussion

Changes in the surface relief of layered GaS and GaS: Yb single crystals caused by  $\gamma$ quanta were also traced by the microscopic (AFM) method. As an example, Fig. 1 shows threedimensional (d) images of the surface of the original (Fig.1a and 1b) and  $\gamma$ -irradiated with 140rad (Fig. 1c, 1d) samples of GaS and GaS:Yb, respectively. Comparison of the surfaces of the initial single crystals shows that if the surface of gallium sulfide is characterized by the presence of sub-roughness and heterogeneity, the introduction of ytterbium impurity into the GaS structure leads to smoothing and uniformity of its surface. In this case, the depth of irregularities in the case of GaS:Yb increases by a factor of ~ 5 (from 8 to 40 nm) compared to GaS. Irradiation of GaS and GaS: Yb single crystals with a dose of 140 krad is accompanied by a change in the surface state of these samples. After irradiation with  $\gamma$ -rays with a dose of 140 krad, the surface state of GaS deteriorates significantly, while minor changes occur on the GaS:Yb surface. Annealing the samples at a temperature of  $t = 100^{\circ}C$  for 1 hour partially restores the surface of the irradiated samples. It should be noted that the analysis of 3d images of surfaces of y-irradiated GaS and GaS:Yb samples with doses of 30,50,100,140 and 200 krad allows us to conclude that the boundary values of the dose of surface changes are 50 and 140 krad, respectively.



Fig.1. Three-dimensional images of the surface of the original (a, b) and  $\gamma$ -irradiated dose with of 140 krad (c, d) of GaS (a, c) and GaS:Yb (b, d) single crystals.

Figures 2 and 3 show the histograms of 2d images (curves of the distribution of surface images by the size of irregularities) in the horizontal and vertical directions of the selected section  $(100\times100 \text{ nm})$ . As can be seen from the histograms, the GaS single-crystal is characterized by an uneven distribution of irregularities, both in the horizontal and in the vertical directions with different heights of ~ (30-40 nm) and a frequency of ~ 16 nm (Fig. 1a and b). GaS:Yb single crystal histograms show a uniform distribution of irregularities in both horizontal and vertical directions with the same height of ~ 25 nm and a frequency of ~ 13 nm (Fig.2a and b). Irradiation with a dose of 140 krad of these samples leads to a strong (Fig.2 c, d) and minor (Fig. 2c, d) changes in histograms for GaS and GaS:Yb, respectively.



Fig. 2. Histograms of 2d images of the initial (a, b) and  $\gamma$ -irradiated doses of 140 krad (c, d) of GaS single crystals in horizontal (a, c) and vertical (b, d) directions.



Fig. 3. Histograms of 2d images of the initial (a, b) and the  $\gamma$ -irradiated dose of 140 krad (c, d) GaS: Yb single crystals in the horizontal (a, c) and vertical (b, d) directions.

Considering the sub-roughness profile in the framework of multifractal analysis, we can assume that it has the invariance property when the same unit free volume (straight cone of height h and diameter d) is continuously repeated over the entire area.

The dose dependence of a single free volume of irregularities (a single average volume of a relief-forming cone), which is shown in Fig. 4, was studied. The dependence is exponential:  $V=A*e^{kx}$ , (V is a single average volume of a relief-forming cone, A is a single crystal constant, k is an absorption coefficient, x is the degree of irradiation). As can be seen from fig. 4, with an increase in the  $\gamma$ -irradiation dose to 140 krad, the value of a single average volume of the relief-forming cone V decreases by ~ 2.5–5 times (from 6000 and 5500 to 2500 and 1000 nm<sup>3</sup>) for GaS and GaS: Yb, respectively.



Fig.4. Dose dependences of the free volume of irregularities in GaS (1) and GaS: Yb (2) single crystals.

#### 4. Conclusion

The surface relief of undoped GaS and doped GaS: Yb single crystals subjected to gamma irradiation was studied by atomic force microscopy (AFM). It was found that GaS is characterized by an uneven distribution of irregularities with different heights ~ (30-40 nm) and a frequency of ~ 16 nm subjected to gamma irradiation. When doping crystals with Yb atoms, the distribution of irregularities become more orderly, the height is ~ 25 nm, and the periodicity of ~ 13 nm irregularities decrease. Irradiation with GaS gamma quanta at doses of  $\Phi_{\gamma}$  <140krad and doping with Yb atoms lead to rearrangement of defects, resulting in a decrease in a single free volume of irregularities, and doses above  $\Phi_{\gamma}$ > 140 krad an increase occurs. The use of a single average volume of a relief-forming cone was introduced as a characteristic of the degree of irradiation on a single average volume of the relief-forming cone are proposed, expressed in the exponential form V = A \* e^{kx}. A correlation was established between the distribution profile of a single free volume of irregularities (a single average volume of a relief-forming cone), obtained by AFM the radiation resistance of layered GaS: Yb single crystals.

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# АСМ-МИКРОСКОПИЯ ПОВЕРХНОСТИ ГАММА-ОБЛУЧЕННЫХ СЛОИСТЫХ МОНОКРИСТАЛЛОВ GaS И GaS:Yb

#### Ф.Г. Асадов

**Резюме:** Впервые методами атомно-силовой микроскопии (ACM) и ИК-Фурье спектроскопии получена информация о рельефе поверхности нелегированных GaS и легированных монокристаллов GaS:Yb, подвергнутых гамма-облучению. Установлено, что для GaS характерно неравномерное распределение неровностей с различной высотой и периодичностью, а при легировании кристаллов атомами Yb распределение неровностей упорядочится, высота и периодичность неровностей уменьшается. В ИК-Фурье спектрах наблюдаются изменения коэффициентов отражения поверхности монокристаллов GaS и GaS:Yb в зависимости от дозы гамма-облучения ( $\Phi_{\gamma}$ =30-200 крад) и на основе этих изменений установлено, что легированные монокристаллы являются более радиационно-стойкими.

*Ключевые слова:* слоистые монокристаллы GaS и GaS:Yb, рельеф, *γ*-облучение, поверхность, спектры отражения, дефект, АСМ-микроскопия

## QAMMA ÜALARLA ÜALANDIRILMI GaS V GaS:Yb LAYLI MONOKRİSTALLARININ SƏTHİNİN AQM MİKROSKOPİYASI

#### F.Q. Əsədov

*Xülasə:* İlk dəfə olaraq atom-qüvvə mikroskopu (AQM) və Furye-İQ-spektroskopiya metodu ilə qamma şüalanmış GaS və legirə olunmuş GaS:Yb monokristallarının səth relyefi haqqında informasiya alınmışdır. Müəyyən edilmişdir ki, GaS üçün səthin müxtəlif hündürlüklü və periodiklikli qeyri-bərabər paylanması, kristalların YB atomları ilə legirə olduqda isə qeyri-bərabərliklərin paylanması nizamlanır, hündürlük və periodiklik isə azalır. Furye-İQ spektrlərdə qamma şüalanmanın dozasından asılı olaraq ( $\Phi_{\gamma}$ =30-200 krad) GaS və GaS:Yb atomlarının səthinin əksolma əmsallarında dəyişiklik müşahidə olunur və bunun əsasında müəyyən olunmuşdur ki, legirə olunmuş monokristallar radiasiyaya daha çox davamlıdır.

Açar sözlər: GaS və GaS:Yb laylı monokristalları, relyef, qamma-şüalanma, səth, əksolma spektrləri, defekt, AQM-mikroskopiya