



Energetic spectrum and some properties of lead sulfide implanted with oxygen

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Abstract

Spectral dependencies of optical reflection and absorption coefficients in lead sulfide implanted with oxygen ions and annealed in vacuum have been investigated at $T = 300$ K. It was found that the average value of hole concentration within the sample space area where properties were modified by ion implantation and vacuum annealing was equal to $(3.25 \pm 0.30) \times 10^{18} \text{ cm}^{-3}$. The depth of the space in question was estimated and its quantity was shown to make the tenths of micrometers. It was demonstrated that because of annealing process oxygen ions occupied places in the chalcogen sublattice healing anion vacancies. It was also found that vacuum annealing of lead sulfide with implanted oxygen did not cause elimination of all anion vacancies. Moreover, the concentration of sulfur vacancies increased considerably in comparison with its value in the initial samples non-subjected to ion implantation. This fact testifies that oxygen in lead sulfide possesses acceptor action which is compensated by chalcogen vacancies. It was established that in the lead sulfide, the only quasi-local energy level, being located in the valence band at the energy distance of 0.16 eV from its top, was connected with oxygen impurity. No other energy level which one could connect with oxygen or with the complexes containing oxygen in lead sulfide was revealed. The storage stability of properties of investigated material was demonstrated.

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1. Introduction

Lead sulfide (PbS) is widely used in optoelectronics for fabricating infrared radiation sources and detectors. PbS-based light-sensitive cells are, as a rule, *n*-PbS polycrystalline layers grown on glass substrates and annealed in an oxygen-containing environment at 500–550°C. Layer annealing is accompanied by a conductivity inversion from *n*- to *p*-type in near-surface crystalline layers and an occurrence of photosensitivity

(see Refs. [1–4] and their respective citations). Even though objects of this type have been studied for more than half a century, the role of oxygen in photoconductivity formation, and its energy spectrum in lead sulfide remain as yet unexplained. This is primarily caused by the complex morphology of oxygen-annealed polycrystalline layers that complicates investigating their properties as well as interpreting the obtained data.

An alternate method of oxygen doping for modifying the properties of lead chalcogenides (and PbS in particular) is currently used besides annealing in an oxygen-containing environment. It is the method of ion implantation combined with post-implantation vacuum

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annealing of layers for 1–2 h at 250–300°C [5,6]. This is also accompanied by a conductivity inversion from *n*- to *p*-type in near-surface layers of oxygen-implanted films and an occurrence of photosensitivity. It appears that ion implantation may be successfully used as a tool of studying the energy spectrum of oxygen in lead chalcogenides. This problem was solved for lead telluride (PbTe) and lead selenide (PbSe) in Refs. [6,7]. These works also demonstrate that in these materials, oxygen forms a single energy level located deep in the valence band and higher than the anion vacancy level. No other energy level that could be connected to oxygen, or oxygen-containing complexes, was found either in PbTe, or in PbSe.

The authors of Ref. [6] also attempted to obtain experimental data on the properties of oxygen-implanted and vacuum-annealed lead sulfide. They demonstrated that the average hole concentration, $\langle p \rangle$, in PbS : O⁺ within the region whose properties had been modified by means of ion implantation and annealing was $(4.0 \pm 0.6) \times 10^{18} \text{ cm}^{-3}$, while the oxygen energy level E_O was located deep in the valence band in 0.16 eV from its upper edge (from here onward oxygen ion-implanted and vacuum-annealed samples will be denoted shortly as *n*-PbS : O⁺ or *p*-PbS : O⁺ with the first letter indicating the conductivity type in the initial non-implanted sample).

These studies have not been concluded and were resumed only 15 years later. They set a number of goals, with the foremost being determining the stability of PbS : O⁺ properties over time. For this purpose a repeat study of optical reflectivity (*R*) spectra in ion-implanted samples was necessary. In case PbS : O⁺ properties were indeed proved to be stable, a detailed study of optical absorption spectra α in PbS : O⁺ would be worth carrying out to estimate the main parameters of the ion-implanted region, in particular its thickness d^* .

Hereinafter the following notations for the thickness of the examined samples will be used for convenience: d_{st} for an original sample with *p*-type conductivity, d_p and d_n for ion-implanted and annealed samples with *p*- and *n*-type conductivity, respectively.

2. Experimental procedure

2.1. Preparing the samples for examination

The procedure of preparing the samples was briefly described in Ref. [6]. We provide a more detailed description below.

The objects of this study were monocrystalline lead sulfide samples grown using the Bridgman–Stockbarger

method and slow cooling from the melt and subjected to homogenized annealing in vacuumed quartz reservoir tubes for a 100 h at 600°C. The monocrystals were doped by either intrinsic crystal lattice defects or a sodium impurity and super-stoichiometric lead (Pb_{ex}).

Free carrier concentrations in studied samples were determined using the Hall method at 78 K. Since considerably large inhomogeneities in the spatial distribution of free carriers are typical for lead sulfide monocrystals, only samples with layer concentrations of free carriers with no more than 3–5% deviation from the Hall values, as determined by optical reflectivity measurements, were chosen for the experiment.

Optical surfaces necessary for examining the reflectivity coefficient spectra were created by mechanically grinding and polishing the samples using the procedure detailed in Ref. [8]. These sample surfaces served as basic and had no further mechanical treatment. These were the surfaces implanted with oxygen using the Vesuvius-1 setup (ion energy was 150 keV, ion-current density was 0.2 $\mu\text{A}/\text{cm}^2$, the dose of implanted oxygen ions was 3000 $\mu\text{C}/\text{cm}^2$, which is equivalent to an oxygen surface concentration $N_0 = 1.87 \times 10^{16} \text{ cm}^{-2}$). The doping process was completed by annealing the implanted samples in quartz reservoir tubes (vacuumed to a pressure of 0.13 Pa) for 2 h at 300°C.

Thin samples necessary for recording optical absorption spectra were prepared through grinding their reverse (non-implanted) sides and subsequently making optical surfaces of them according to the procedure described in Ref. [8].

2.2. Measurement procedure

The reflectivity (*R*) and the transparency (*T*) coefficients for all samples were measured point by point at room temperature. The number of spectral passes varied from 20 to 30 for *R* measurements and from 100 to 180 for the *T* ones. The obtained values of R_i and T_i for each fixed wavelength λ_i were averaged. The scatter of isolated points in the reflectivity coefficient spectra was no more than 1–2%, and that in the transparency coefficient spectra was an order of magnitude less. The $R(\lambda)$ and $T(\lambda)$ dependencies obtained in such a manner were smoothed and used to calculate the absorption coefficient by the formula:

$$\alpha = (1/d) \ln \frac{(1-R)^2 + [(1-R)^4 + 2R^2 T^2]^{1/2}}{2T}. \quad (1)$$

The thickness d of the studied samples was measured using a MP-4 microinterferometer with a reliability of $s = 0.90$.

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