PHYSICS AND CHEMISTRY
OF SOLID STATE
V. 27, No. 2 (2026) pp. 361-365

Section: Physics

DOI: 10.15330/pcss.27.2.361-365

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ФІЗИКА І ХІМІЯ ТВЕРДОГО ТІЛА
Т. 27, № 2 (2026) С. 361-365

Фізико-математичні науки

PACS: 61.72.Cc; 66.30.Lw

ISSN 1729-4428 (Print)
ISSN 2309-8589 (Online)

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Surface effects in neutron-irradiated CdSb crystals

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The influence of structural defects in fast neutron-irradiated CdSb to $2 \cdot 10^{18}$ n/cm^2 on the state of the crystal surface was studied. It was shown that during storage of irradiated crystals, their surface (layer 60 ÷ 80 μm) increases its conductivity. A model is proposed that explains the influence of irradiation on the state of the crystal surface. The migration energy of interstitial cadmium atoms, the values of diffusion coefficients at different temperatures, and the frequency factor are experimentally determined. Also, the isochronal annealing of radiation defects in neutron-irradiated CdSb crystals was studied in detail.

Keywords: surface, radiation defects, diffusion coefficients, neutron-irradiated, annealing, CdSb crystals.

Received 22 December 2026; Accepted 19 June 2026; Published 27 June 2026.

Introduction

The concentration of radiation defects exceeds the concentration of uncontrolled impurities at high doses of irradiation and the role of radiation defects in the formation of the physical properties of crystals becomes dominant. Often, when irradiated crystals are stored, their characteristics and properties change over time. Studies of the spatial distribution of radiation defects (formed during storage of irradiated crystals) indicate that they may be concentrated in the surface layer of the crystal.

The annealing process was [1-4] and is [5-9] part of a set of measurements to establish the nature and mechanisms of the physicochemical properties of semiconductor materials. Theoretical approaches to annealing are based on the kinetics of defect reactions – this is the diffusion of interstitial and vacancy components, their mutual recombination or the formation of more stable complexes. Important concepts are the mobility of defects (diffusion coefficients), activation energies of migration, capture/emission of charge carriers by defects, as well as kinetic models of isochronous and isothermal annealing.

The proceedings [10] contain 30 papers. The topics discussed include: activation energies for vacancy migration, clustering and annealing in silicon; vacancy defects in III-nitrides; determination of defect content and defect profile in semiconductor heterostructures; detection of vacancy-like defects during Cu diffusion in GaAs by positron annihilation, etc.

In [11] the infrared spectra of room-temperature, neutron-irradiated Si were investigated. During annealing, the 827 cm^{-1} VO defect band decreases, and another band at 885 cm^{-1} generally attributed to the VO$_2$ centre increases. The kinetics of the evolution of these two defects was investigated. In work [12] was established that in order to obtain the optimal values of thermoelectric figure of merit Z_a in n-Si crystals doped with nuclear transmutation, it is necessary to anneal them at 1100 – 1200 °C for 2 h.

Based on swelling data and microstructural observations, the low dose neutron irradiation-induced defects in silicon nitride-based ceramics were considered to be primarily point defects [13]. In order to investigate the kinetics of defect recovery, these irradiated specimens were isothermally and isochronally annealed continuously up to 1473 K. The isochronal and isothermal annealing behaviors of the irradiated SiC were investigated in [5]. Invisible point defects and defect clusters are found to be the dominating defect types in the neutron-irradiated SiC, and the amount of defect recovery reaches a maximum value after isothermal annealing for 30 min.

The authors [14] have related the results of mechanical spectroscopy, transmission electron microscopy, electrical resistivity, differential thermal analysis and small angle neutron scattering, as obtained in deformed and neutron irradiated Mo single crystals. For isochronous annealing of radiation defects after neutron irradiation of GaP, process parameters were obtained [7].

Currently, very few studies have been published on the explanation of defect formation in neutron-irradiated (at high doses) CdSb crystals. As a result, the mechanisms of radiation defect formation and their nature are poorly understood. Therefore, it is advisable to develop a model interpretation of the influence of defects on the surface state of irradiated CdSb crystals.

I. Materials and Method

The annealing of radiation defects was studied in neutron-irradiated CdSb crystals (NI-CdSb). The crystals were irradiated with fast reactor neutrons to 2×10^{18} n/cm^2 ($E = 2$ MeV). The irradiation temperature did not exceed 340 K. Slow neutrons were cut off with a cadmium filter. To reduce radioactivity to a safe level, the samples were kept in hot chambers for 5-8 months.

The electric field was applied along the crystallographic direction [010]. Before measurements, the crystals were polished with a micron diamond paste and etched in a mixture of concentrated H_2O_2, HF(70 %), HCl and glycerin (10:3:1:6). This was done to remove the surface layer, which in many cases could affect the results of volumetric studies.

II. Result and discussion

Neutron irradiation of undoped *p*-CdSb crystals (see Fig. 1a, curve 1) to 2×10^{18} n/cm^2 leads to a conversion of the conductivity type (from *p*-type to *n*-type), forming donor-type defects with an ionization energy of $E_C - 0.16$ eV (Fig. 1a, curve 2). When storing NI-CdSb crystals, their resistance measured at nitrogen temperature decreased over time, while at room temperature the resistance remained practically unchanged. This is explained by the fact that at room temperature, due to the ionization of deep donors $E_C - 0.16$ eV, the crystal passes into a low-resistance state, which masks the effects associated with shallow donors at low temperatures. The study of the spatial distribution of shallow donors (formed during storage of irradiated crystals) showed that they are mainly concentrated in the surface layer of the crystal with a thickness of $60 \div 80$ μm [15]. Surface etching (by $60 \div 80$ μm) led to an increase in the crystal resistance. This is the result of removing the surface lower-resistance defect region that formed during crystal storage. Storage of the etched crystal at $T \geq 270$ K again led to the formation of a surface, low-resistance layer. This behavior of NI-CdSb can be explained by the fact that storage of irradiated crystals leads to the appearance in their volume of mobile donor centers ionized at nitrogen temperature. After formation, they diffuse to the crystal surface, thereby reducing the resistance of the surface region.

The beginning of the process of diffusion of donor centers to a freshly etched surface (which can be considered as an unsaturated drain) is well described by an exponential dependence (see Fig. 1b):

$$\frac{R}{R_0} = exp(-k\tau) \qquad (1)$$

This indicates that when the surface is far from being saturated with defects, the process of diffusion of defects from the volume to the surface is monomolecular in nature. After a certain time interval, which depends on the storage temperature of the crystal, the surface is saturated with defects, and the equation of diffusion of defects ceases to have an exponential character (see Fig. 1b).

Also, the process of diffusion of defects to the crystal surface does not depend on the type of chemical reactions that can occur between atoms on the crystal surface and the surrounding environment. The conducted studies have shown that the nature and speed of the process of changing

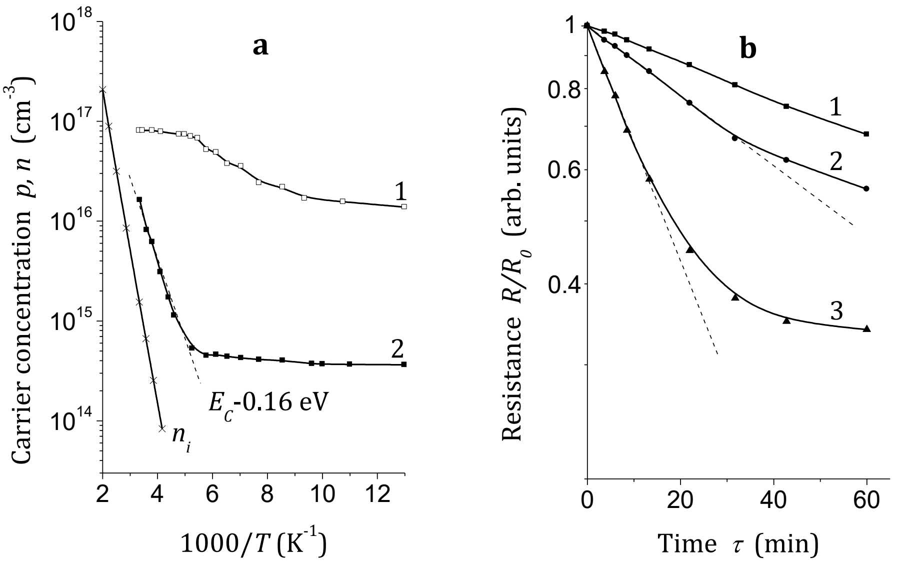

Fig. 1. (a) Carrier concentrations as a function of temperature for CdSb: 1 is unirradiated; 2 is neutron-irradiated. (b) Resistance at 77 K for a neutron-irradiated with freshly etched surface CdSb vs. storage time at temperatures *T*: 1 – 273 K; 2 – 293 K; 3 – 318 K.

the resistance of irradiated crystals during storage practically does not depend on the atmosphere in which the irradiated crystal was located (in air, in alcohol or in glycerin). The phenomenon of diffusion of defects from the volume to the crystal surface was not observed in unirradiated, both in pure and in doped with various impurities crystals. It had a weakly pronounced character in γ-irradiated crystals (obviously, due to the small concentration of radiation defects) [16, 17].

Fig. 2a shows the dependences of the time during which the resistance of the freshly treated surface decreased by a factor of $e = 2.7$ (in the exponential interval) on the storage temperature of the irradiated crystal. The activation energy of the process determined from this dependence using [18]:

$$ln\,\tau_0 = ln\,C + \frac{E}{kT} \qquad (2)$$

is equal to (0.36 ± 0.03) eV. Obviously, this energy is the energy of defect migration, which is formed during the decay of defect clusters in NI-CdSb. From the curve in Fig. 1b, for the exponential interval of the dependence of the resistance on time, we can determine the value k (Eq. 1) and, accordingly, the coefficient of defect diffusion D at different temperatures ($k = AD$ [18]). When determining the coefficient A, it was assumed that the sink for defects is the surface of the crystal in the form of a parallelepiped, far from saturation.

The linear dependence of lgD on $1/T$ shown in Fig. 2b allows us to conclude about the activation nature of the diffusion coefficient D, which can be represented by the equation:

$$D = D_0 \cdot exp\left(\frac{E}{kT}\right) \qquad (3)$$

The energy value determined from the slope of dependence $D(T)$ is equal to (0.37 ± 0.02) eV, which agrees well with the value of E determined from the analysis of Arrhenius curves (Eq. 2) and confirms the correctness of the previously determined value of the migration energy of point radiation defects in NI-CdSb.

An important parameter of diffusion processes is the value D_0, which is sometimes called the frequency factor. The value D_0, determined from our experimental data (using Eq. 3) is equal to $\approx 10^3$ cm^2/s.

Fig. 3 shows the isochronal annealing curves of NI-CdSb. Annealing was carried out in a muffle furnace with a uniform temperature field distribution in a nitrogen atmosphere. Annealing lasted 10 min at each temperature. Before measuring the resistance of the crystals, their surface layer 60 ÷ 80 μm was removed. This was done in order to obtain information about the annealing of defects in the crystal volume. The resistance of the annealed crystals was measured at two temperatures – 77 K (Fig. 3a) and 293 K (Fig. 3b). At $T = 77$ K, the crystal resistance is determined by shallow ionized centers. Deep donor levels $E_C - 0.16$ eV capture electrons and have little effect on the resistance at low temperatures. As can be seen from Fig. 3a, with the annealing of the irradiated crystal, its resistance measured at $T = 77$ K, slowly decreases in a wide temperature range up to 100 °C. We believe that at this stage of annealing, the breakdown of defect clusters (clusters formed by neutron irradiation). In this case, the lattice is enriched with shallow (ionized at $T = 77$ K) donor-type defects, which reduce the crystal resistance at low temperatures. These defects can exit from the volume to the crystal surface, creating a low-resistance surface layer. The migration energy determined by us earlier for such defects is (0.36 ± 0.03) eV. Shallow donor-type defects may be due to interstitial Cd atoms (Cd$_i$). In this case, it is most likely that the defect clusters formed upon neutron irradiation are clusters of interstitial cadmium atoms. The annealing of defect clusters occurs over a wide temperature range [1], which is consistent with the annealing of Cd$_i$ clusters in NI-CdSb. The formation of clusters of interstitial cadmium atoms upon irradiation with high-energy particles has been observed in CdS crystals [2]. Clusters of interstitial atoms have also been identified in silicon crystals [3].

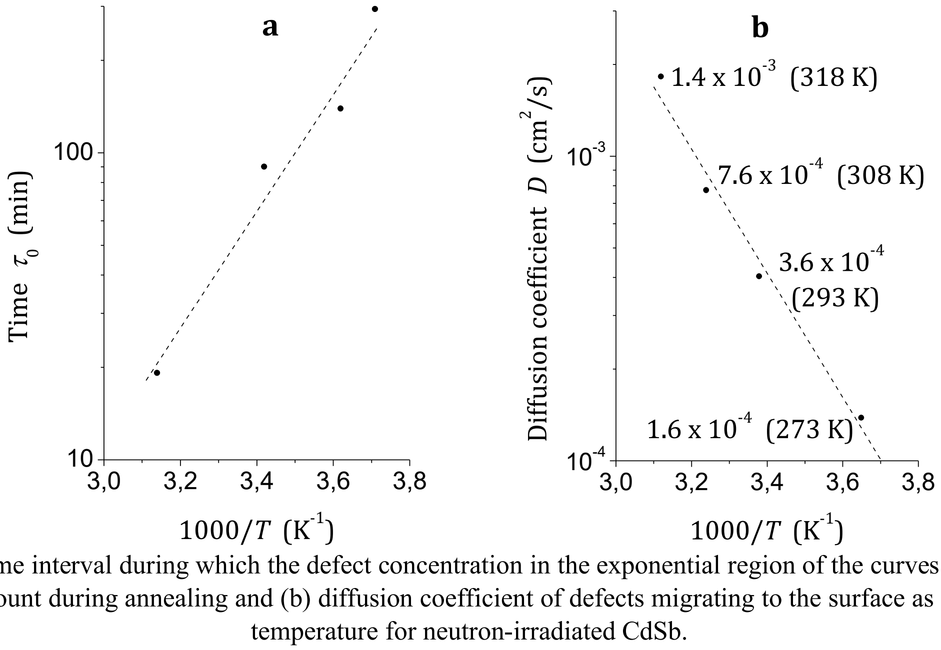

Fig. 2. (a) Time interval during which the defect concentration in the exponential region of the curves decreases by the same amount during annealing and (b) diffusion coefficient of defects migrating to the surface as a function of temperature for neutron-irradiated CdSb.

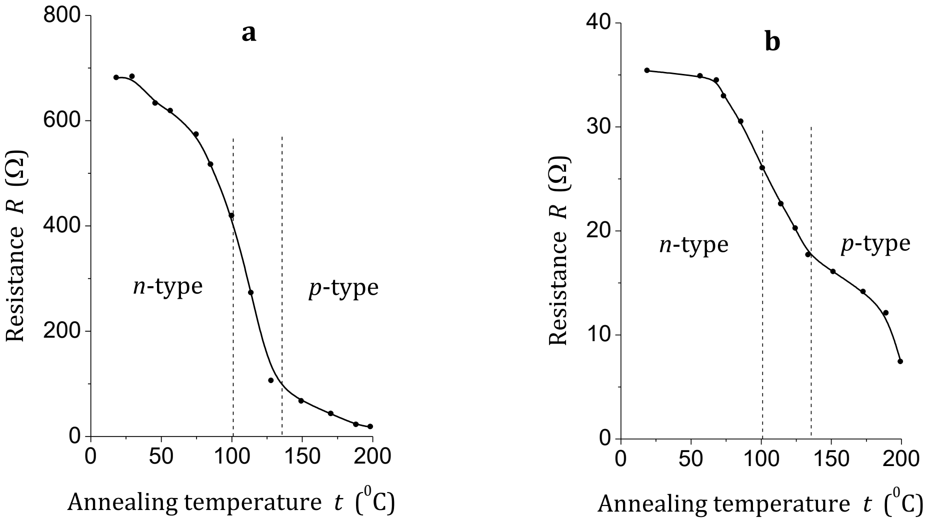

Fig. 3. Isochronal annealing of defects in neutron-irradiated CdSb. Measurement of crystal resistance was carried out at (a) $T = 77$ K and (b) $T = 293$ K. Annealing at each temperature lasted 10 min.

The resistance of the irradiated crystal measured at room temperature practically does not change during annealing to ~ 80 °C and decreases sharply at higher annealing temperatures (see Fig. 3b). At room temperatures, the resistance of the crystal is determined by deep donor levels $E_C - 0.16$ eV, the concentration of which in NI-CdSb is dominant. With a constant concentration of deep donors, the formation of shallow donors Cd_i (a consequence of the decay of clusters) will have little effect on the resistance of the crystal at room temperature, except for its slight decrease with increasing annealing temperature (see Fig. 3b).

Starting from the annealing temperature of 80 °C (see Fig. 3b), annealing of deep donors with $E_C - 0.16$ eV occurs. Obviously, during annealing of deep donors, shallow acceptors ionized at nitrogen temperature are formed. As a result, the resistance of the crystal decreases and its conductivity type changes. In the transition region of annealing, the conductivity type was not identified. After annealing at 130 °C, the irradiated crystals had *p*-type conductivity, as before irradiation. The sharp decrease in the crystal resistance at the measurement temperature of 77 K (see Fig. 3a) in the annealing interval of 100 ÷ 120 °C is obviously conditional not only with the formation of shallow acceptors (appearing during the annealing of deep donors), but also with an increase in the mobility of free carriers (due to the annealing of scattering centers of radiation origin). Also, the influence of mobility on conductivity is mainly significant at low temperatures, where the mobility under irradiation undergoes the largest change. NI-CdSb annealed at 200 °C became similar in electrical properties to undoped *p*-CdSb crystals.

The deep donor $E_C - 0.16$ eV formed after neutron irradiation may be a complex consisting of a cadmium vacancy (V_{Cd}) and an uncontrolled impurity. The decay of such a complex releases V_{Cd}, which is a shallow acceptor. Complexes of radiation-induced defects consisting of cation vacancies and uncontrolled impurities (Cl, Te, S) are well identified by EPR in ZnSe [4]. Such complexes in ZnSe are annealed at temperatures above 100 °C.

Conclusions

The influence of structural defects in NI-CdSb on the surface state of crystals has been established. It is shown that during storage of irradiated crystals, their surface (layer 60 ÷ 80 μm) increases its conductivity. A model is proposed that explains the irradiation effect on the surface state of crystals. The migration energy of Cd_i, the values of diffusion coefficients at different temperatures, and the frequency factor have been experimentally determined.

The annealing of radiation defects in NI-CdSb was investigated. Defects responsible for conductivity at 77 K are annealed in a wide temperature range up to ~ 130 °C. According to the model proposed by us, at this stage of annealing, the Cd_i clusters disintegrate. Defects responsible for deep donors $E_C - 0.16$ eV begin to anneal at a temperature of 80 °C. At the same time, the lattice is enriched with shallow acceptors. At the annealing stage of 100 ÷ 130 °C, the conductivity type changes from *n*- to *p*-type. It is assumed that complexes containing cadmium vacancies and uncontrolled impurities are responsible for deep donors. Deep donors $E_C - 0.16$ eV at 77 K are fast recombination centers.

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Поверхневі ефекти в кристалах CdSb, опромінених нейтронами

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Досліджено вплив структурних дефектів в CdSb, опроміненому швидкими нейтронами до 2×10^{18} н/см^2, на стан поверхні кристала. Показано, що під час зберігання опромінених кристалів їхня поверхня (шар 60 ÷ 80 мкм) збільшує свою провідність. Запропоновано модель, яка пояснює вплив опромінення на стан поверхні кристала. Експериментально визначено енергію міграції міжвузлових атомів кадмію, значення коефіцієнтів дифузії при різних температурах та частотний фактор. Також детально досліджено ізохронний відпал радіаційних дефектів у кристалах CdSb, опромінених нейтронами.

Ключові слова: поверхня, радіаційні дефекти, коефіцієнти дифузії, опромінення нейтронами, відпал, кристали CdSb.