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David C. Look

Wright State University - Main Campus, david.look@wright.edu

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High-temperature annealing in electron-irradiated CdS[†]

David C. Look

Department of Physics, University of Dayton, Dayton, Ohio 45469
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The room-temperature irradiation of as-grown *n*-type CdS by 1-MeV electrons results in an increase in carrier concentration. The damage anneals in the temperature range 250–300 °C, and a study of the annealing kinetics reveals an activation energy of about 1.3 eV which is attributed to sulfur-vacancy motion.

It is known that room-temperature bombardment of CdS by electrons, ions, x rays, or neutrons results in lattice damage which affects electrical, optical, and thermal properties. Some of this damage is stable at room temperature and anneals only after heating above 200 °C^{1–4}; however, to our knowledge, there have been no previous studies of annealing kinetics at these temperatures. In this paper we describe isothermal-annealing studies on electron-irradiated CdS, using Hall-effect measurements. The discussion will be limited to two representative crystals, characterized by initial carrier concentrations, $N_0 = 10^{15}$ and 10^{17} cm⁻³, respectively. Consider a typical bulk *n*-type CdS crystal with $N_0 \approx 10^{15}$ cm⁻³. Upon electron bombardment at 1 MeV we expect to create S and Cd interstitials and vacancies (S_i , Cd_i , V_s , V_{Cd} , respectively).² Since the electron concentration N is observed to increase to greater than 10^{16} cm⁻³ (after bombardment by about 10^{17} electrons), we assume that donors are being created faster than acceptors. (Another possibility, the destruction of acceptors, could not account for an increase greater than the initial concentration of acceptors, about 10^{15} cm⁻³.) There is good evidence that the interstitials are quite mobile at 25 °C⁵ and thus they probably either annihilate with the vacancies or else move away to be trapped by sinks, such as dislocations.⁶ After the irradiation, then, the dominant electrical defect should be the donor vacancy, i. e., the V_s .⁷ The vacancies in II-VI compounds are expected to be stable at 25 °C,⁸ although it has been conjectured that under a sufficiently strong attraction the V_s can move a few lattice distances, so that V_s -impurity donor complexes are possible.⁹

After bombardment by more than about 10^{17} electrons, N reaches a saturation value of about 2×10^{16} cm⁻³ and, in fact, begins to slowly decrease with further irradiation. This is probably due to the fact that above a certain V_s concentration a newly created S_i is almost certain to annihilate a V_s before it is trapped at a sink. However, the V_{Cd} acceptors will continue to increase slowly, causing N to decrease. It should be noted that in a chlorine-doped sample (15 ppm) with $N_0 > 10^{17}$ cm⁻³, N was observed to decrease at all levels of bombardment. This could be due to the room-temperature Fermi level being above the created donor energy level in this sample.

When the irradiated crystal is heated sufficiently above 25 °C, annealing can take place in several ways: (i) the isolated V_s can become mobile and find the trapped S_i ; (ii) the trapped S_i can break away and find the V_s ; (iii) either mobile defect, but especially the V_s might undergo temporary trapping at impurities or other defects before annihilation. If (i) is the dominant process we might expect a kinetic equation of the form¹⁰

$$\frac{dn_i}{dt} = -\nu_0 \exp\left(-\frac{E}{kT}\right) zv(n_v^0 - n_i^0 + n_i)n_i, \quad (1)$$

where n_v (n_v^0) and n_i (n_i^0) are the instantaneous (initial) concentrations of vacancy and trapped interstitials, respectively, ν_0 is the jump frequency, and E the activation energy of vacancy motion, v is the volume per lattice site, and z is the number of vacancy sites, surrounding a trapped interstitial, from which a single jump toward the interstitial means certain annihilation. For Eq. (1) to be valid the V_s concentration in the immediate vicinity of the trapped S_i must be the same as the average concentration in the whole crystal.⁶ Although this is certainly not rigorously true at all times we will still use Eq. (1) as a first approximation. For $n_i \gg n_v^0 - n_i^0$, the solution to Eq. (1) gives a "second-order" reaction:

$$n_i = n_i^0 [1 + n_i^0 \nu_0 zv \exp(-E/kT)t]^{-1}. \quad (2)$$

Thus, a plot of n_i^0/n_i vs t should have a slope $\approx n_i^0 \nu_0 zv \exp(-E/kT)$. For an order-of-magnitude estimate of $\nu_0 zv$ we may take $\nu_0 \approx 10^{13}$ sec⁻¹, $z \approx 10$, and $v \approx 10^{-22}$ cm³ to get $\nu_0 zv \approx 10^{-8}$. If we assume that there are few trapped S_i in the sample before irradiation, and that one new carrier is produced for each new S_i (or each new V_s), then $N - N_0 \approx n_i$, where, again, N_0 is the carrier concentration before irradiation.

FIG. 1. Additional carrier concentration, $N - N_0$, caused by a room-temperature electron bombardment, as a function of annealing time at 250, 275, and 300 °C.

FIG. 2. Additional carrier concentration at the beginning of the anneal, $N(0) - N_0$, divided by the instantaneous value, $N(t) - N_0$, as a function of annealing time at 250, 275, and 300 °C. See text for explanation of plot.

In Fig. 1 are plotted 250, 275, and 300 °C isothermal annealing data for a sample of approximate dimensions $10 \times 4 \times 1$ mm³, grown by vapor-phase deposition, and doped with about 20 ppm fluorine. The annealing was carried out in an argon atmosphere. For this sample, $N_0 = 4 \times 10^{15}$ cm⁻³, and these are typical annealing data for samples of approximately this concentration.

In Fig. 2 is plotted $[N(0) - N_0]/[N(t) - N_0]$ vs t and it is seen that each isothermal annealing curve has a region in which it approximately obeys second-order kinetics, i.e., Eq. (2). The activation energy can be determined by comparing slopes at two different temperatures. In this way we get $E_{250-275} \approx 1.5$ eV, $E_{250-300} \approx 1.4$ eV, and $E_{275-300} \approx 1.3$ eV, and $\nu_0 z v \approx 10^{-6} - 10^{-8}$, depending upon temperature. Since $\nu_0 z v$ is approximately what we estimated earlier, and since the calculated E 's are typical for vacancy motion,⁶ we conclude that our model is reasonable.

Toward the end of each anneal, when $n_i \leq (n_0^0 - n_i^0)$, Eq. (1) predicts an approach to "first-order" kinetics, i.e., $dn_i/dt \propto n_i$, leading to an exponential decrease in $N - N_0$. This appears to be happening, as seen in Fig. 1, for $N - N_0 \leq 10^{15}$ cm⁻³, but calculations of E and $\nu_0 z v$ in this range are somewhat unreliable because of uncertainty in N_0 . At the beginning of each anneal the more rapid variation of $N - N_0$ with t is probably due to higher-order exponentials which are often found in diffusion-limited processes.⁶

The variation of E with temperature would be expected if the vacancies were subject to temporary trapping, say at impurities.¹¹ In this connection it is interesting

to note that V_g -halogen complexes have been proposed before as an explanation of certain electrical properties in CdS.¹² However, low-temperature electrical measurements have not confirmed this situation in our sample.

Additional light is shed on these matters by means of a quenching experiment, in which an unirradiated sample was heated above 325 °C, the temperature at which N begins to increase rapidly. Subsequent cooling to 300, 275, or 250 °C, and monitoring of N vs t , reveals annealing kinetics very similar to those in irradiated samples. If the dominant process for annealing after irradiation were S_i breaking away from traps, above about 250 °C, then we would expect that for annealing after quenching the S_i would not undergo significant trapping at 300 °C and we would observe an activation energy characteristic of the more mobile interstitial motion, i.e., perhaps about 0.1 eV. Since the same E 's are observed in both cases we conclude that they are probably characteristic of vacancy motion and that the interstitials remain trapped at 300 °C.

Another standard method of data analysis is to use the relationship¹³

$$\frac{d(N - N_0)}{dt} = -A(N - N_0)^n \exp(-E/kT). \quad (3)$$

where A is a constant and n is the order of the reaction. For $(N - N_0) \approx 4 - 6 \times 10^{15}$ cm⁻³, this gives $n \approx 1.7 - 2.2$ for all temperatures and E within 0.1 eV of the values obtained before. Thus, second-order kinetics seems to be a reasonable assumption over this range of $N - N_0$.

If vacancy-impurity complexes are playing a role in the annealing, then we would expect this role to be less important at higher temperatures.¹¹ Thus, the values $E \approx 1.3$ eV and $\nu_0 z v \approx 10^{-8}$ are probably most indicative of the "bare-vacancy" motion. Further experiments are in progress to elucidate this matter.

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