

Hydrogen-related complexes formed by electron-irradiation of hydrogenated silicon

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We studied point defects, vacancies (V), and self-interstitials (I), formed by room-temperature electron irradiation of hydrogenated Si via the observation of the optical-absorption spectra due to complexes formed of hydrogen and point defects. The formation of H_2^* , a metastable state of H_2 in Si, was also studied. Specimens were n -type, p -type, and high-purity Si crystals. They were doped with hydrogen (H) by annealing at 1300 °C in H_2 gas followed by quenching. Then they were irradiated with 3 MV electrons at room temperature. We measured their optical-absorption spectra by a Fourier-transform infrared spectrometer at about 7 K. Many optical-absorption peaks were observed. Among them, 1987 and 1990 cm^{-1} peaks were weak in B-doped specimens and the 2122 cm^{-1} peak was weak in P-doped specimens. These results support the hypothesis that the 1987 and 1990 cm^{-1} peaks and the 2122 cm^{-1} peak are due to complexes including I and V , respectively. The H_2^* concentration does not show good correlations with the concentrations of V and I . We hypothesize that H_2^* is formed from the interaction between an H_2 and a Frenkel pair.

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I. INTRODUCTION

Many studies¹ have been performed on the optical-absorption spectra due to hydrogen (H)-point defect complexes in proton implanted Si,² Si grown in H_2 gas,^{3,4} and electron⁵ or neutron irradiated⁶ Si grown in H_2 gas or hydrogenated Si.^{7,8}

One of the issues concerning H-point defect complexes in Si is the identification of point defects that are included in complexes responsible for optical-absorption peaks at 1987 and 1990 cm^{-1} , and 2122 cm^{-1} (the wave numbers are rounded off at the first decimal point). Many models have so far been proposed.^{2,4-6,9,10} Among them, the model of Bech Nielsen *et al.*² seems most reliable since they performed *ab initio* calculation of vibrational frequencies of H in various states to explain their experimental results of optical-absorption spectra of proton implanted Si crystals. Combining their experimental and calculational results, they proposed that the 2122 cm^{-1} peak and the 2223 cm^{-1} peak are due to $V \cdot H_2$ and $V \cdot H_4$ (V : a vacancy) complexes, respectively, and tentatively proposed that the 1987 and 1990 cm^{-1} peaks are due to $I \cdot H_2$ (I : a self-interstitial).

The numbers of H atoms included in those complexes were almost conclusively determined from the splitting of those peaks due to coimplantation of H and D . On the other hand, species of point defects were identified only by the above calculation. Suezawa¹¹ sought to experimentally determine them by experiment alone. He focused on the effects of impurities on the concentration of intrinsic point defects, I and V , in electron-irradiated Si, and found that intensities of the 1987 and 1990 cm^{-1} peaks were much weaker in p -type Si and C-doped Si than those of intrinsic Si, both of which were irradiated with electrons at RT after hydrogenation. This result strongly supports the hypothesis that the 1987 and 1990 cm^{-1} peaks are due to $I \cdot H_2$ since the I concentration is known to be low in p -type Si and C-doped Si, which is consistent with the findings of Bech Nielsen *et al.*² The first purpose of the present study was to systematically investigate the dependence of the generation of $I \cdot H_2$ on various

specimens to more quantitatively confirm the above identification. In this context, further experimentation is needed to show the incorporation of V in the 2122 cm^{-1} defect. A V is known to form a pair with group V donors such as P. Hence, the concentration of isolated V is expected to be low in P-doped Si, probably resulting in a smaller concentration of $V \cdot H_2$ in P-doped Si. The second purpose was to study the dependence of the generation of $V \cdot H_2$ on various specimens.

One issue concerning the interaction between point defects and H_2 is the formation mechanism of H_2^* , a metastable state of H_2 , from H_2 in electron-irradiated Si. Optical-absorption peaks due to H_2^* were observed in proton implanted Si crystals¹² at about 817, 1838, and 2168 cm^{-1} . Subsequently, such peaks were also found by Newman *et al.*⁷ in electron-irradiated Si doped with H_2 . They proposed a formation mechanism of H_2^* from H_2 in which an H_2 is transformed into H_2^* by capturing a V and then an I or vice versa. A subsequent study by Estreicher *et al.*¹³ of the model of Newman *et al.* by *ab initio* calculation supported the existence of this mechanism. It intuitively seems that the formation process of H_2^* in proton implanted Si may be different from that of electron-irradiation of hydrogenated Si since hydrogen atoms are in an atomic state in the former case and are in a molecular state in the latter case. In a previous paper,¹⁴ we experimentally studied the formation of H_2^* due to electron-irradiation of hydrogenated Si and concluded that combinations of an H_2 and a V or of an H_2 and a Frenkel pair were the formation mechanism of H_2^* from the interaction between an H_2 and electron-irradiation induced point defects. This hypothesis is not consistent with that proposed by Newman *et al.* and Estreicher *et al.* According to their model, an isolated V and an isolated I equally participate in the formation of H_2^* . According to our experimental results, however, the formation rates of H_2^* in B-doped and C-doped Si crystals were similar to those in high-purity specimens. Thus, an isolated I seems to play a small role, if any, since the intensity of the $I \cdot H_2$ peak was found to be very weak in B-doped and C-doped Si crystals as mentioned

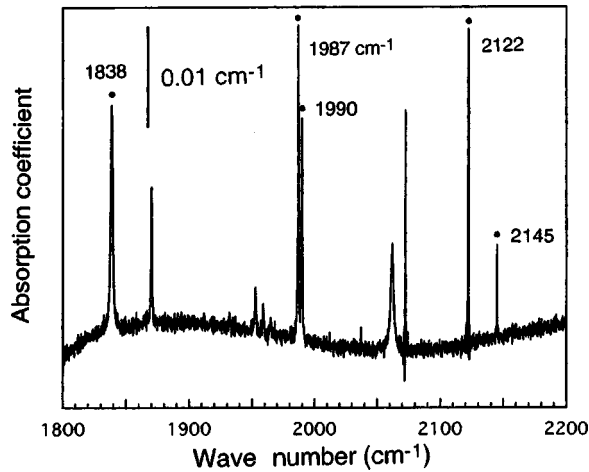


FIG. 1. The optical-absorption spectrum of electron-irradiated Si doped with H around 2000 cm^{-1} . The irradiation dose was $2 \times 10^{16}\text{ cm}^{-2}$.

above. The third purpose of this study was to clarify the role of V in the formation of H_2^* in various specimens.

II. EXPERIMENT

Specimens were high-purity (n -type; P concentration of about $4 \times 10^{12}\text{ cm}^{-3}$), n -type ($1.5 \times 10^{16}\text{ P}$ and $2.4 \times 10^{15}\text{ P}\cdot\text{cm}^{-3}$, abbreviated as PH and PL, respectively, hereafter) and p -type ($1.5 \times 10^{16}\text{ B}$ and $2.6 \times 10^{15}\text{ B}\cdot\text{cm}^{-3}$, abbreviated as BH and BL, respectively) floating-zone grown Si crystals. Specimens were cut from the above crystals with a diamond slicer. After being cut, they were shaped with carborundum and chemically etched with a mixed acid of $\text{HNO}_3:\text{HF} = 3:1$. The specimen size was about $6 \times 6 \times 11\text{ mm}^3$. To dope the specimens with H, they were sealed in quartz capsules together with H_2 gas. They were heated at $1300\text{ }^\circ\text{C}$ for 1 h followed by quenching in water. Most hydrogen atoms are in a state of H_2 in quenched specimens since hydrogen atoms form pairs during quenching to reduce the solubility that depends on the specimen temperature. Specimens were irradiated with 3 MV electrons at RT. During irradiation, the specimens were pressed onto a sample holder that was cooled with running water and blown air. To ensure that damage was nearly uniform throughout the 6-mm-thick specimens in the case of electron irradiation, each sample was irradiated equally on both sides. The total dose indicated in Sec. III was the sum of that irradiation. Optical-absorption spectra of those specimens were measured with a Fourier-transform infrared (FTIR) spectrometer and a continuous flow-type liquid-helium cryostat. The resolution was 0.25 cm^{-1} .

III. RESULTS

A. General

Figure 1 shows a part of the optical-absorption spectrum of electron-irradiated Si doped with H_2 . Observed absorption peaks were as follows: 2145, 2122, 2072, 2062, 1990, 1987, 1953, 1952, 1870, 1838, 1600, 817, and 719 cm^{-1} . Peak

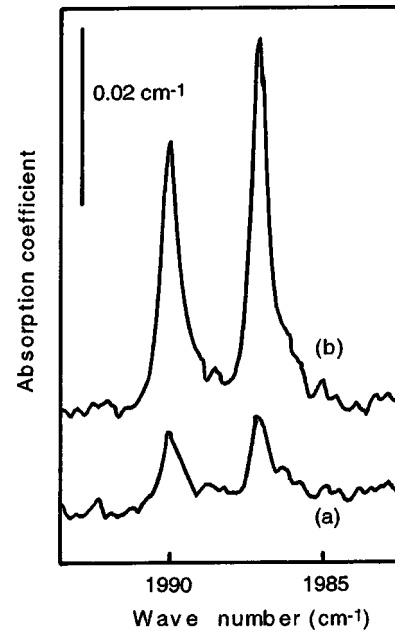


FIG. 2. Optical-absorption spectra of (a) BH and (b) high-purity specimens around 1990 cm^{-1} . The electron dose was $1.2 \times 10^{17}\text{ cm}^{-2}$.

positions of most absorption peaks agree well with those observed in proton implanted Si.² As mentioned in Sec. I, we herein mainly discuss peaks at 1838, 1987, and 1990, and 2122 cm^{-1} . The line widths at half maximum (abbreviated as FWHM, hereafter) of the 1838, 1987, and 2122 cm^{-1} peaks are about 1.40, 0.63, and 0.27 cm^{-1} , respectively. As a measure of defect density responsible for those peaks, we plot the peak intensity rather than the areal intensity in the following.

B. The 1987 and 1990 cm^{-1} peaks

Figure 2 shows the optical-absorption spectra of (a) a BH specimen and (b) a high-purity specimen after electron-irradiation of $1.2 \times 10^{17}\text{ cm}^{-2}$. The spectra have been shifted along the ordinate for the sake of simplicity. The intensities of the 1987 and 1990 cm^{-1} peaks in the BH specimen were much weaker than those in the high-purity specimen. This result is interpreted to be due to the lower concentration of I in B-doped Si than that in high-purity Si because of the change of substitutional B to interstitial B by I in the former crystal. Hence, this result clearly shows that these two peaks are due to an $I\cdot\text{H}_2$ complex, not to $V\cdot\text{H}_2$. The most remarkable point of this result is that we can detect I by the fact that it forms a complex with H_2 . This will be discussed in Sec. IV. Figure 3 shows the dependence of the 1987 cm^{-1} peak intensity on the electron dose in various specimens. Generally, the peak intensity increases linearly at low doses and then is saturated or decreases at high doses. Comparing the peak intensities of the BL specimens and those of the high-purity specimens, we can estimate the efficiency of capture of I by B as compared with that of H_2 as follows. The peak intensity of the 1987 cm^{-1} peak at a dose of about $0.2 \times 10^{17}\text{ cm}^{-2}$ is about $4 \times 10^{-2}\text{ cm}^{-1}$ and that of the BL

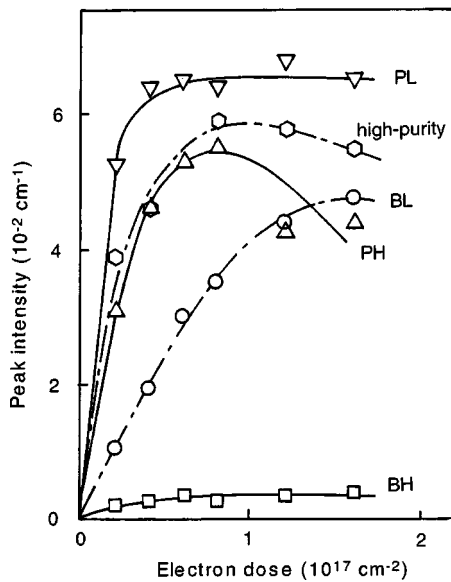


FIG. 3. The dependence of the 1987 cm^{-1} peak intensity on the electron dose in various specimens.

specimen is about $1 \times 10^{-2} \text{ cm}^{-1}$. Hence, the decrease of peak intensity due to B doping is $3 \times 10^{-2} \text{ cm}^{-1}$. Since the H_2 concentration is estimated to be about $1.7 \times 10^{16} \text{ cm}^{-3}$ from the doping condition, the relative efficiency of the capture of I is given as $(3 \times 10^{-2} / 1 \times 10^{-2}) \times (1.7 \times 10^{16} / 2.6 \times 10^{15}) \sim 20$.

C. The 2122 cm^{-1} peak

Figure 4 shows the optical-absorption spectra of (a) a high-purity specimen and (b) a PH specimen after electron irradiation of $1.2 \times 10^{17} \text{ cm}^{-2}$. The intensities of the 2122 and 2145 cm^{-1} peaks in the PH specimen were much weaker than those in the high-purity specimen. These results are interpreted to be due to the lower concentration of V in the PH specimen than that in the high-purity specimen because of the formation of a $V \cdot P$ pair in the former specimen. Hence, these results clearly show that the 2122 and 2145 cm^{-1} peaks are due to $V \cdot \text{H}_2$, not to $I \cdot \text{H}_2$. According to Bech Nielsen *et al.*,² the 2122 and 2145 cm^{-1} peaks are due to $V \cdot \text{H}_2$ with different modes. Figure 4 shows the dependence of the 2122 cm^{-1} peak intensity on the electron dose in various specimens. Generally, again, the peak intensity increases linearly at low doses and then is saturated or decreases at high doses. Comparing the peak intensities of the PH specimen and the high-purity specimen at a dose of about $1 \times 10^{17} \text{ cm}^{-2}$, we notice that the efficiency of the capture of V by P is about 5 times higher than that of H_2 in high-purity specimens.

Combining the results shown in Figs. 2–5, we can experimentally show that both V and I in electron-irradiated Si are detected via their interaction with H_2 . Figure 6 shows the dependences of the peak intensities of the 1838, 1987, 2122, and 2145 cm^{-1} peaks at low doses. The peak intensities of the latter three are proportional to the electron dose. Incidentally, the concentration of defects responsible for an absorption peak is given by multiplication of the areal intensity,

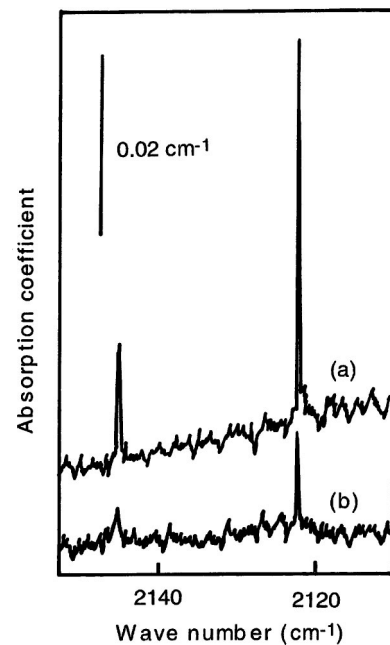


FIG. 4. Optical-absorption spectra of (a) high-purity and (b) PH specimens around 2120 cm^{-1} . The electron dose was $1.2 \times 10^{17} \text{ cm}^{-2}$.

which is proportional to multiplication of peak intensity and the FWHM, and the conversion factor that is inversely proportional to the oscillator strength. If we assume that the multiplications of the FWHM and the conversion factors for the 1987 and 2122 cm^{-1} peaks are the same and that the capture cross sections of H_2 for I and V are the same in high-purity Si, the generated concentrations of V and I due to electron-irradiation are almost the same at low doses. This agrees well with our intuition, but such results have never been shown experimentally. Hence, our method to detect I and V via their complexes with H_2 is expected to open up a way of investigating radiation damage in semiconductors as well as in Si.

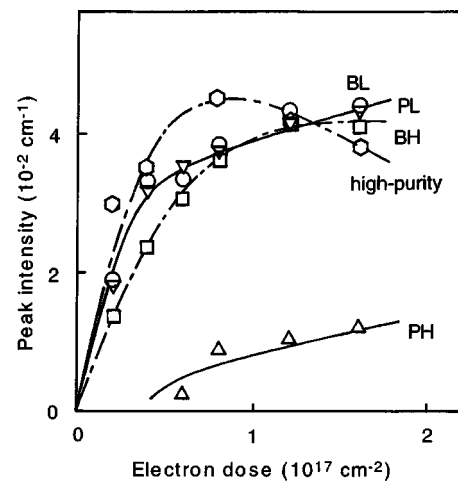


FIG. 5. The dependence of the 2122 cm^{-1} peak intensity on the electron dose in various specimens.

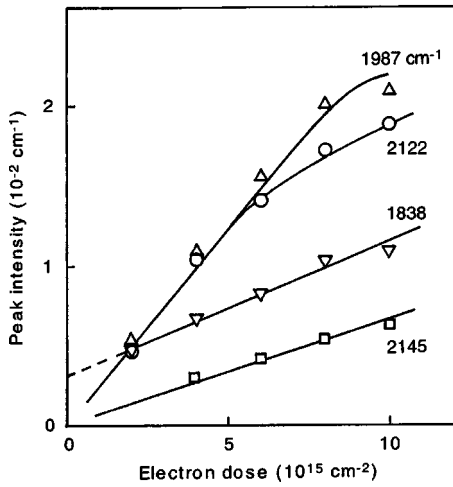


FIG. 6. The dependences of the 1838, 1987, 2122, and 2145 cm^{-1} peak intensity on the electron dose at low doses in high-purity specimens.

D. The 1838 cm^{-1} peak

Next we show the results for H_2^* . In the previous paper,¹⁴ we proposed two possible mechanisms by which H_2^* is formed from the interaction between H_2 and electron-irradiation-induced damages, namely, capture of a V or a Frenkel pair by an H_2 . These proposed mechanisms were based on the dependence of H_2^* concentration on the electron dose in high-purity, B-doped (BH) and C-doped specimens. Figure 7 shows the results. The ordinate is the 1838 cm^{-1} peak intensity. Comparing Fig. 7 with Figs. 3 and 5, it can be noticed that the dependences in Fig. 7 are much different from those of $I \cdot \text{H}_2$ and $V \cdot \text{H}_2$: the dependence of the intensity of H_2^* on

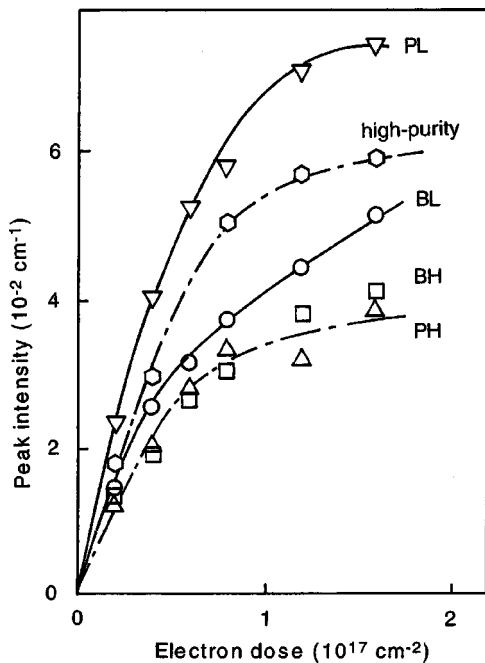
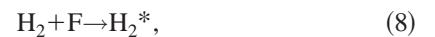
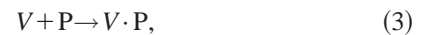


FIG. 7. The dependence of the 1838 cm^{-1} peak intensity on the electron dose in various specimens.

specimens is not so strong as those of $I \cdot \text{H}_2$ and $V \cdot \text{H}_2$. This means that only an I or only a V cannot form H_2^* by interacting with H_2 in Si. Hence, in the sense that both I and V are necessary to form H_2^* , the model of Newman *et al.*⁷ and Estreicher, Hastings, and Fedders¹³ is supported. The above results, however, as already suggested in the previous paper,¹⁴ deny the possibility that isolated V and I form H_2^* by separately combining with H_2 since the formation rate of H_2^* does not strongly depend on the specimens, and also because the formation rate linearly depends on the electron dose at low doses as shown in Fig. 6. (The intercept at dose 0 in Fig. 6 corresponds to the quenched in intensity.¹⁵) The concentrations of isolated I and V are proportional to the electron dose and the formation rate of H_2^* should be proportional to the square of electron dose if H_2^* is formed by an H_2 combining separately with an isolated I and V . Hence, we hypothesize that the formation of H_2^* by electron irradiation of a hydrogenated Si crystal is due to the interaction between an H_2 and a Frenkel pair, which is a much more restricted situation than that postulated by the model of Newman *et al.* and Estreicher, Hastings, and Fedders. The transformation process of H_2 to H_2^* after the capture of a Frenkel pair is probably the same as that proposed by Estreicher, Hastings, and Fedders. According to Fig. 6, the formation rate of H_2^* is about $\frac{1}{3}$ those of $I \cdot \text{H}_2$ and $V \cdot \text{H}_2$ under assumptions similar to those made in Sec. III C. This may be interpreted to mean that the concentration of the Frenkel pair is about $\frac{1}{3}$ those of I and V . If this interpretation is correct, the above result is very interesting since we can estimate the concentration of the Frenkel pair. A Frenkel pair probably dissociates into V and I or is annihilated by recombination itself or with V and I if it is not captured by H_2 . These probabilities are expected to strongly depend on the irradiation temperature. Experiments using a low-temperature irradiation method should thus be conducted.

IV. DISCUSSION

First, we discuss the reactions of electron-irradiation-induced defects (V , I , and Frenkel pair) among themselves as well as with impurities. The following reactions are possible:



where Si, Bi, and F mean a Si atom at a normal lattice site, B at an interstitial site, and a Frenkel pair, respectively. During

electron-irradiation at RT, V , I , and F can move very fast in the specimen. Here, we neglect other possible reactions such as $I+F$, $V+F$, and so on. Moreover, we do not take into consideration higher-order reactions, for example, $V \cdot H_2 + V \rightarrow V_2 \cdot H_2$, which probably become dominant at high doses and lead to the saturation or decrease of peak intensities shown in Figs. 3 and 5. We first assume that the concentrations of I , V , and F do not depend on the specimens just after irradiation. In other words, the primary knockon events do not depend on the specimens. Reactions (1), (2), (4), (5), (7), and (8) commonly occur in high-purity, B-doped and P-doped specimens. Even for the same reaction, for example, reaction (1), there may be a difference of reaction efficiency depending on the specimens, since some defects may have appropriate charge states depending on the dopant concentration. Reactions (3) and (6) occur in P-doped and B-doped specimens, respectively. In the following, we will not discuss reactions (2) and (5) since we detected optical absorption due to V_2 only in an n -type specimen because a V_2 is optically active only in a neutral charge state and we did not detect an optical-absorption peak due to I_2 . Incidentally, comparing Figs. 3 and 5, it can be noticed that the $I \cdot H_2$ intensities at high doses are roughly 1.5 times larger than those of $V \cdot H_2$. This may be interpreted as being due to the greater ease of V_2 formation as compared with I_2 formation.

As already estimated, the efficiency of reactions (3) and (6) are about 5 and 20 times larger than those of reactions (4) and (7), respectively. The efficiency is determined by capture cross sections of B, P, and H_2 for moving I and V . To determine the magnitude of a capture cross section, we need to know the nature of the interaction between defects and impurities, namely, if it is long range or short range. The Coulombic interaction and the interaction via the strain field are representative of long-range interactions, the former being of longer range. Without electron irradiation, B and P are negatively (B^-) and positively (P^+) charged, respectively, at RT. Intuitively, the charge states of B and P are greatly distributed during electron irradiation. The above results, however, suggest that B and P are ionized to B^- and P^+ during electron-irradiation and strongly interact with I and V , respectively. An I and a V probably act as a donor and an acceptor resulting in I^+ and V^- , respectively. Hence, the interactions between I and B and also between V and P are long-range Coulomb interactions resulting in a large capture radius. On the other hand, H_2 is probably in a neutral charge state. Hence, the interactions between I or V and H_2 are probably not Coulombic. Both I and V form complexes with H_2 , i.e., $I \cdot H_2$ and $V \cdot H_2$, respectively, with similar formation rates in high-purity specimens as shown in Fig. 6. This means that the interactions between I or V and H_2 are not long-range, strain-field-type interactions, but rather short-range ones that act only within a limited atomic distance. This hypothesis is based on the opposite strain field around I and V , namely, dilatational and compressive, respectively. Hence, the capture radius around H_2 is small.

Here, it should be remembered that the magnitude of the capture cross section does not correspond to the magnitude of binding energy. For example, the capture cross section of

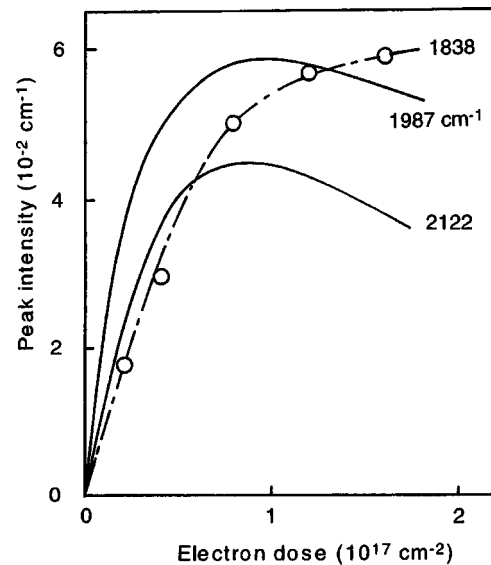


FIG. 8. The dependence of the 1838, 1987, and 2122 cm^{-1} peak intensity on the electron dose in high-purity specimens.

V by H_2 is probably smaller than that of V by P, but the binding energy of $V \cdot H_2$ is larger than that of a $V \cdot P$ pair since the former is thermally more stable than the latter.¹⁶ The origin of the difference of reaction efficiency between I and B and that of V and P is not clear, but it is probably due to the difference of the stability of Bi and the $V \cdot P$ pair that are the products of reactions (3) and (6), respectively. The former forms a pair with B at a substitutional site that is much more stable than the $V \cdot P$ pair.¹⁷ We suppose that some of the $V \cdot P$ pairs formed during electron-irradiation dissociate because of low thermal stability, resulting in a lower efficiency of reaction (3). To confirm this hypothesis, we need to perform low-temperature irradiation where a $V \cdot P$ pair is not dissociated.

Shi *et al.*⁵ observed 1987 and 1990 cm^{-1} peaks in electron-irradiated Si grown in H_2 gas. They proposed that they were due to $V \cdot H_2$ or $V_2 \cdot H_2$. Although their identifications are incorrect as shown above, they succeeded in detecting an I in electron-irradiated Si. According to Fig. 5, the formation rates of $I \cdot H_2$ and $V \cdot H_2$ are similar at low doses. Their formation rates are proportional to the concentrations of constituent defects, I and V , and also to the diffusion rates of I and V , respectively. The above results suggest that the diffusion rates of I and V are similar at RT since the concentrations of I and V generated by electron irradiation are similar. In this context, it should be remembered that the diffusion rate of V during electron irradiation seems much lower than that of I at low temperature, say 20 K, since only V exists after electron irradiation at such a low temperature. The reason for the fast movement of I during irradiation at low temperature is explained by the Bourgoin-Corbett mechanism,¹⁸ which may not work during irradiation at RT. It would be of interest to perform experiments of electron irradiation of hydrogenated Si at low temperature.

For the sake of easy comparison, we plot dependences of peak intensities of the 1838 (H_2^*), 1987 ($I \cdot H_2$), and 2122 ($V \cdot H_2$) cm^{-1} peaks on the electron dose in high-purity speci-

mens in Fig. 8. For the sake of simplicity, we have not plotted the data points of the 1987 and 2122 cm^{-1} peaks. The 1838 cm^{-1} peak intensity shows a much different dependence on electron dose than those of the 1987 and 2122 cm^{-1} peaks at high doses. This suggests that an H_2^* has much smaller capture cross sections for I , V , and the Frenkel pair. This is probably due to the geometrical configuration of H_2^* . As already mentioned, the decreases of peak intensities of the 1987 and 2122 cm^{-1} peaks are probably due to captures of I and V , respectively, at high doses. The formation of larger complexes such as $I_2 \cdot \text{H}_2$ and $V_2 \cdot \text{H}_2$ will be shown in a separate paper.

V. CONCLUSION

We studied optical-absorption spectra of electron-irradiated Si doped with hydrogen and noticed that hydrogen is very useful for detecting vacancies, self-interstitials, and also Frenkel pairs. Experiments on the irradiation of hydrogenated Si at various temperatures should be carried out.

Moreover, extension of our method of optical-absorption measurement after electron irradiation of hydrogenated Si to methods of evaluation employing various techniques of high-energy particle irradiated semiconductors doped with hydrogen should yield fruitful information about irradiation induced defects not yet clearly detected.

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