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Observation of the formation of electron irradiation induced secondary defects in Czochralski silicon

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Electron irradiation and *in situ* observation of Czochralski silicon are carried out in a 2 MeV ultra-high voltage electron microscope at temperatures from room temperature to 400° C. Two kinds of irradiation-induced interstitial type secondary defects are found to be formed; (1) dislocation loops formed in the bulk of the specimen and (2) dislocation loops formed near the electron incident surface of the specimen. The experimental results show that supersaturation of interstitials occurs in the region near the electron incident surface of the specimen during irradiation. It is suggested that some vacancy clusters are formed by irradiation with a high electron flux.

1. Introduction

There have been a number of investigations on the electron irradiation induced secondary defects in silicon [1–10]. It was reported that the secondary defects formed at around 400° C were self-interstitial type dislocation loops lying on {1 1 3} planes, which were unfaulted during continuous irradiation [5, 7]. A large difference in the formation of the defects between float-zone (Fz) silicon and Czochralski (Cz) grown silicon was shown using high voltage electron microscopy by the present authors' group in 1985 [9]. In the case of Fz silicon, there existed a long incubation period of about several minutes for the formation of electron microscopically visible defects and the defects were formed with a localized spatial distribution near the electron incident surface of the specimen. However, in the case of Cz silicon, the defects were formed at around 400° C immediately after the start of irradiation and they had a random distribution in the bulk of the specimen. If a remarkable difference in the oxygen concentration between Cz and Fz silicon crystals is taken into account, it can be suggested that the oxygen atoms play an important role in the secondary defects' nucleation process. The reason why the secondary defects are always formed near the top surface of the Fz silicon specimen after a long incubation time is not yet known. Asahi *et al.* [7] proposed the possibility that this was because some impurities on the surface were shot by electrons and implanted in during the irradiation, which caused the nucleation of the defects.

Between room temperature and 200° C, the induced secondary defects were too small to determine whether they are of interstitial or vacancy type. Furuno *et al.* [6] carried out two-step irradiation experiments at two different temperatures with a 1 MeV high voltage electron microscope. They reported that interstitial loops produced by irradiation at temperatures above 400° C were shrunk by re-irradiation at 200° C, and at the same time new small defects were formed. It was also observed that a re-irradiation above 400° C led to the disappearance of the small defects and the formation of larger interstitial loops. They concluded accordingly that the small defects formed at around 200° C were of the vacancy type. On the other hand, Asahi *et al.* [7] concluded that all of the secondary defects formed in the temperature range between − 100 and 550° C were interstitial loops from the fact that the nucleation process and spatial distribution of the small defects formed below 300° C were quite similar to those of the larger defects formed at the higher temperatures; the nature of the latter being determined as interstitial type by the inside–outside contrast method.

In this study, Cz silicon was irradiated at temperatures between room temperature and 400° C in a 2 MeV ultra-high voltage electron microscope with an electron flux of up to $2 \times 10^{24}\,m^{-2}\,sec^{-1}$. Re-irradiation experiments at different temperatures were also performed. To determine the nature of the small defects formed at lower temperatures, the $2\frac{1}{2}$D method and the inside–outside contrast method were used. By comparing the formation of the secondary defects in the region near the electron incident surface of the specimen and that in the bulk, an inhomogeneous distribution of interstitials in the specimen during the irradiation was suggested.

2. Experimental details

The oxygen and carbon content of the Cz silicon single crystal used in the present experiment were 1.0×10^{18} and 5.0×10^{15} atoms cm^{-3}, respectively. Discs of 3 mm in diameter and 0.5 mm thick with ⟨1 0 0⟩ normal were machined from a single crystal by a diamond wheel cutter and an ultrasonic drill, and then a hollow

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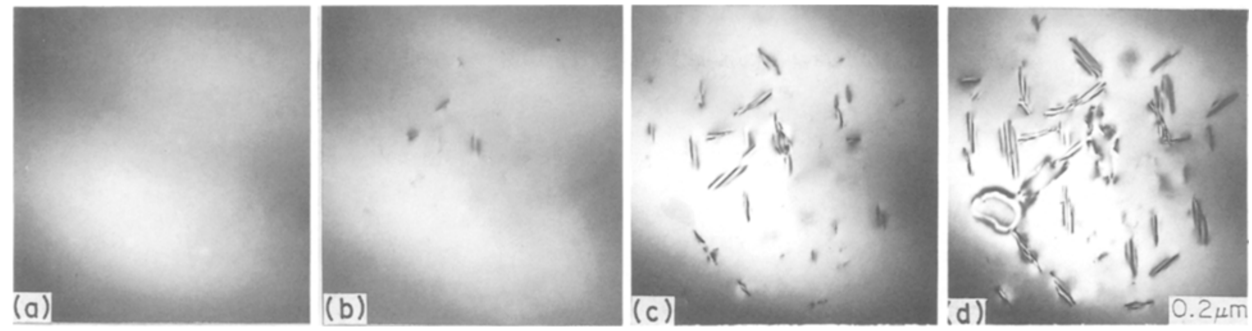

Figure 1 Sequence for irradiation of Cz silicon at 400° C with an electron flux of $2 \times 10^{24}\,m^{-2}\,sec^{-1}$. The thickness of the wedge shape specimen at the centre of the irradiated area is approximately 340 nm. The irradiation times are (a) 61 sec, (b) 128 sec, (c) 175 sec, (d) 220 sec.

of 0.2 mm deep was made at the centre of each disc with the ultrasonic drill. Finally these discs were chemically polished in a mixed solution of nitric acid and hydrogen fluoride until a perforation was made at the centre to obtain a wedge shape around the hole suitable for electron microscopy. Some Fz silicon samples (oxygen: 1×10^{15} atoms cm^{-3}, carbon: 1.8×10^{17} atoms cm^{-3}) in $\langle 1\,1\,1\rangle$ orientation were also used for comparison.

Electron irradiations and *in situ* observations were carried out in the Hitachi HU-3000 ultra-high voltage electron microscope of Osaka University operated at 2 MV. A heating stage on a universal goniometer was used. The electron flux density was measured by inserting a Faraday cage just below the projection lens. The nature of the induced secondary defects and their spatial distributions were investigated in detail in a conventional transmission electron microscope (JEM 200CX) after irradiation.

3. Results

3.1. Irradiation of Cz silicon specimens at elevated temperatures

As was reported in a previous paper [9], when irradiation was carried out at temperatures above 300° C with the electron flux below $10^{24}\,m^{-2}\,sec^{-1}$ in the case of specimens thicker than about 400 nm, interstitial type dislocation loops were observed to form only a few seconds after the start of the irradiation when the first micrograph could be taken. With the dose accumulated, no new loops were nucleated but the existing loops continued to grow except for a small number of them which shrank and finally disappeared. It was found that a prolonged irradiation in which the total electron dose reached about $2 \times 10^{26}\,m^{-2}$ led to the formation of new loops. Stereomicroscopy showed that the new loops were localized in a thin layer near the specimen top surface, i.e., the electron incident surface, while the loops first formed had a random spatial distribution in the bulk of the specimens.

Figure 1 shows a result of the irradiation of a region of 340 nm thick of a Cz silicon specimen at 400° C with a high electron flux ($2 \times 10^{24}\,m^{-2}\,sec^{-1}$). It should be noticed that, as a peculiar phenomenon in this case, no loop can be observed even after 61 sec irradiation, as seen in Fig. 1a. After about 100 sec, corresponding to an electron dose of about $2 \times 10^{26}\,m^{-2}$, loops appeared and grew, as shown in Figs 1b, c and d. Contrary to the former low electron flux case of $10^{24}\,m^{-2}\,sec^{-1}$, all of these loops were found by the stereomicroscopy to be localized near the specimen top surface. By the inside–outside contrast method, they were determined to be of the interstitial type.

In Fig. 2 a typical sequence for the irradiation of Cz silicon crystals of the thickness more than 400 nm with a high flux at temperatures between 300 and 400° C is shown. It can be seen that the loops appear quickly after the irradiation starts. However, as the irradiation is continued, the loops formed, particularly those in the central area where the flux is highest, start to

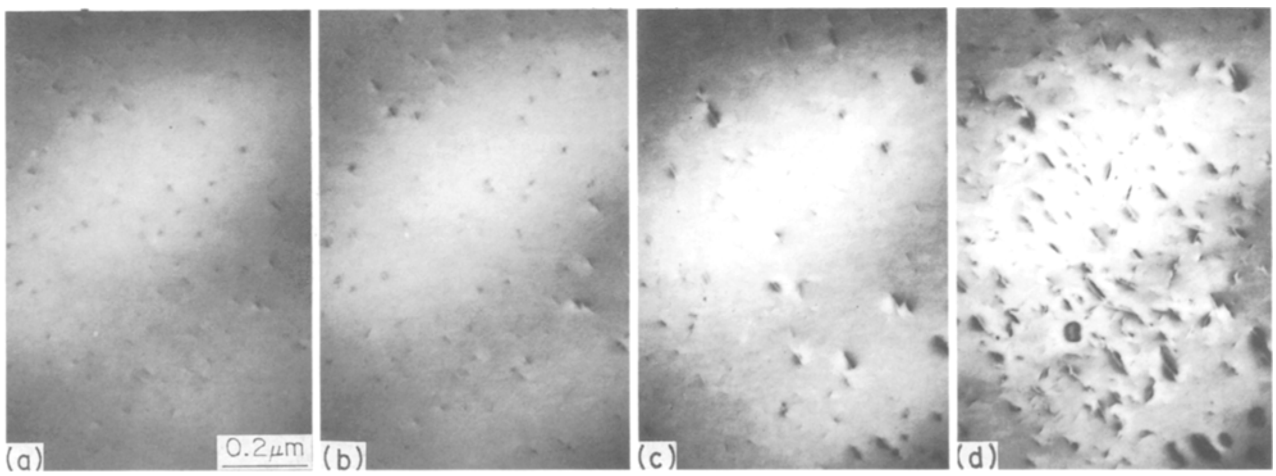

Figure 2 Sequence for irradiation of Cz silicon at 300° C with an electron flux of $1.8 \times 10^{24}\,m^{-2}\,sec^{-1}$. The thickness of the specimen at the centre of the irradiated area is approximately 470 nm. The irradiation times are (a) 30 sec, (b) 52 sec, (c) 129 sec, (d) 210 sec.

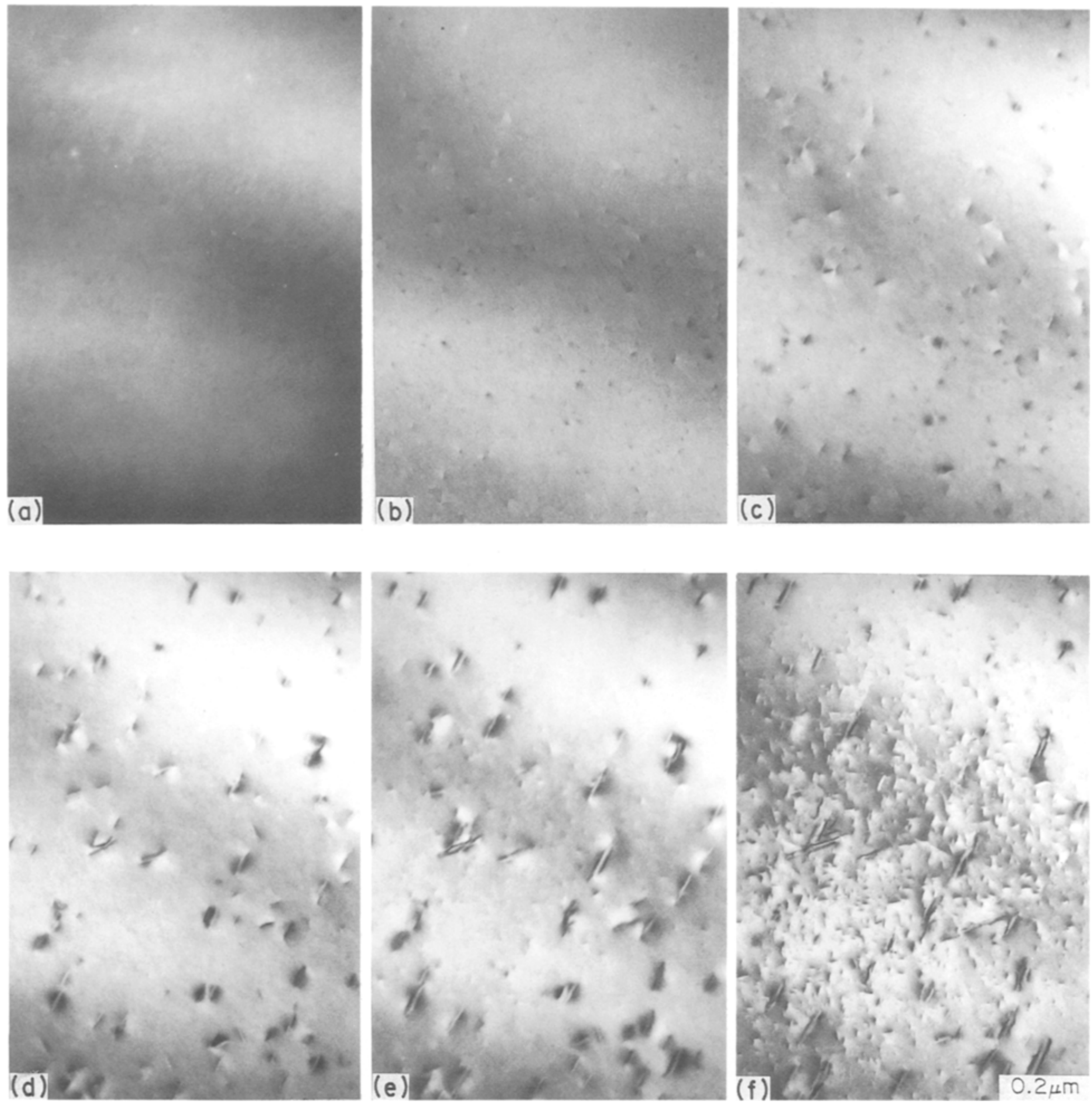

Figure 3 Sequence for irradiation of a 600 nm thick Cz silicon specimen at 250° C. The electron flux is $8 \times 10^{23}\,m^{-2}\,sec^{-1}$. The irradiation times are (a) 10 sec, (b) 33 sec, (c) 88 sec, (d) 211 sec, (e) 369 sec, (f) 580 sec.

shrink and eventually disappear (compare Figs 2a to b). After about 2 min irradiation, corresponding to an electron dose of $2 \times 10^{26}\,m^{-2}$, new loops appear as shown in Figs 2c and d. Stereomicroscopy showed that these new loops were localized near the specimen top surface, while the loops formed in the beginning were situated randomly in the bulk. The new loops were also found to be of interstitial type by the inside–outside contrast method.

3.2. Irradiation of Cz silicon specimens at lower temperatures

Figure 3 shows a typical development of the irradiation induced defects in a specimen thicker than 500 nm at temperatures between 100 and 250° C. In Fig. 3a taken after ony 10 sec irradiation some loops can already been seen as faint contrasts. After 33 sec the defect contrasts become clear and the defect density increases, as shown in Fig. 3b. On further irradiation some of the defects continue to grow but many of them start to shrink and eventually disappear as seen in Figs 3c and d. When the irradiation time reaches about 250 sec, corresponding to a total electron dose of about $2 \times 10^{26}\,m^{-2}$, small dislocation loops showing so-called black–white contrasts appear and their density increases rapidly with further irradiation.

A stereo pair of the defects produced in a specimen 600 nm thick by an irradiation at 180° C for 490 sec with the electron flux of $8 \times 10^{23}\,m^{-2}\,sec^{-1}$ is presented in Fig. 4. It can be seen that the defects which were formed shortly after the start of irradiation and grew to the sizes larger than 40 nm, are randomly distributed in the bulk of the specimen and those formed after a certain incubation period, at smaller sizes of $10 \sim 20$ nm, are situated in a very thin layer within several tens of nanometres from the electron incident surface.

When the irradiation was carried out on regions

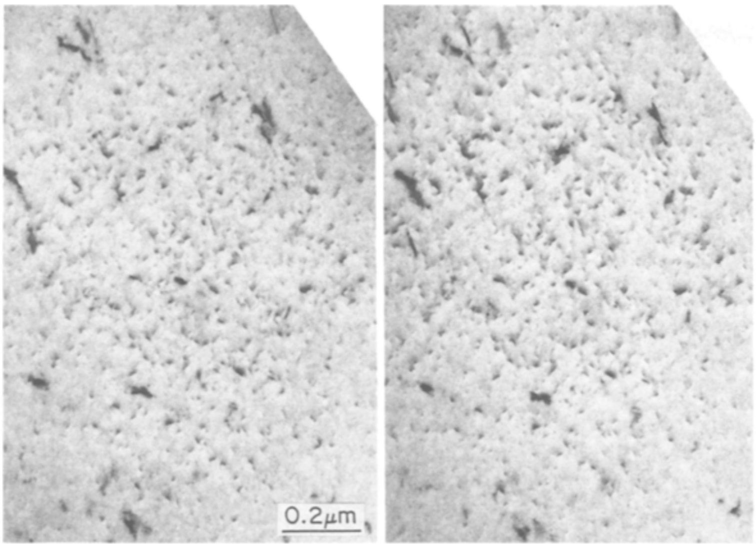

Figure 4 Stereomicrographs showing the spatial distribution of the secondary defects in Cz silicon after 490 sec irradiation at 180° C.

where the foil thickness was below 500 nm, or at temperatures below 100° C, no loops could be observed until the small loops were formed after an electron dose of $2 \times 10^{26}\,m^{-2}$.

3.3. Two-step irradiation at different temperatures

Re-irradiation of Cz silicon specimens at temperatures between room temperature and 200° C after the first irradiation at 400° C were carried out. Figure 5 shows *in situ* observation sequences for re-irradiation at 200, 120 and 40° C, respectively. It can be seen that the loops formed by the first irradiation shrink with the re-irradiation. The shrinkage speed becomes faster as the re-irradiation temperature is decreased from 200 to 40° C, while the incubation time for the formation

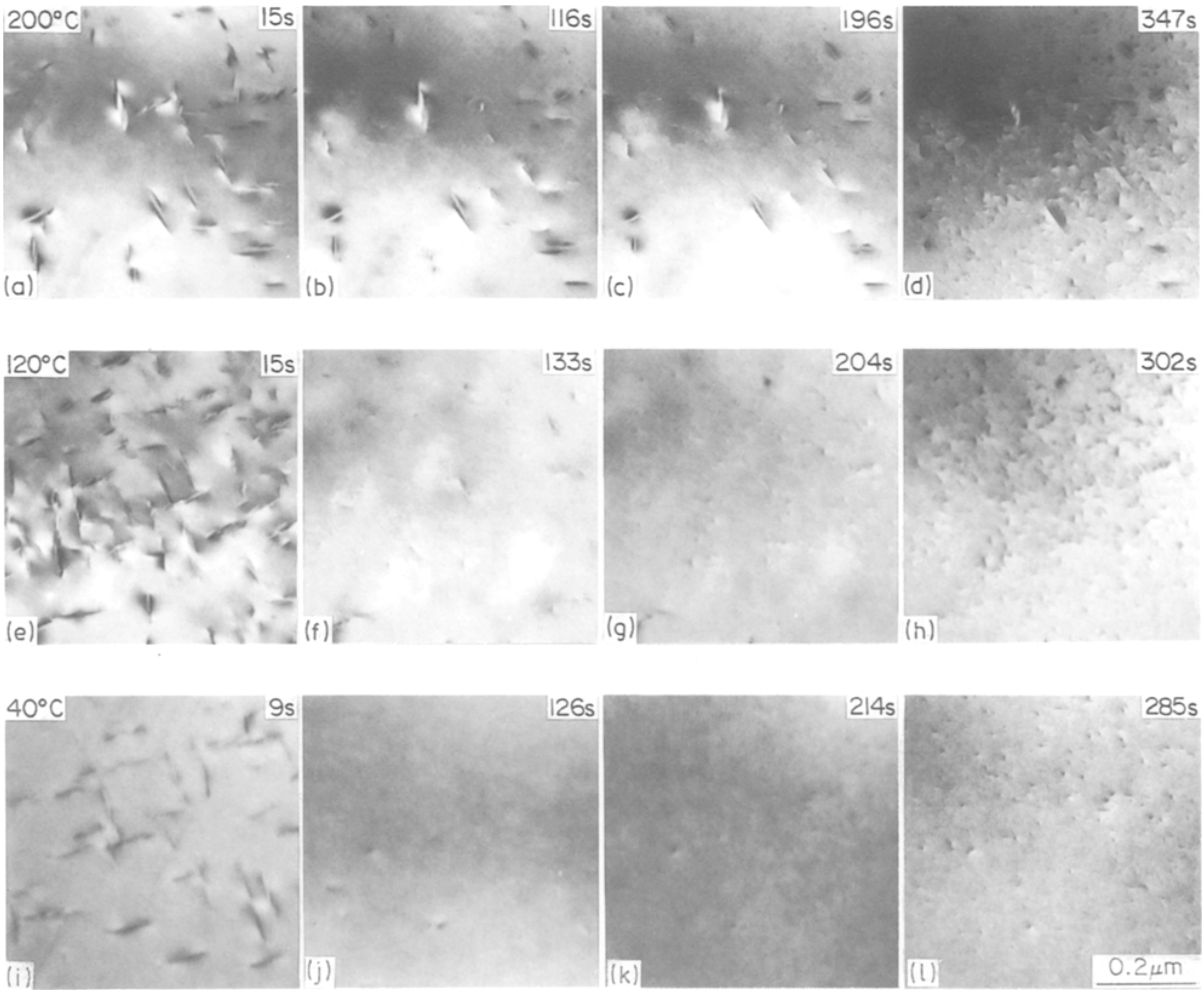

Figure 5 Effect of re-irradiation on Cz silicon at 200, 120 and 40° C, respectively. The specimen was previously irradiated at 400° C. The re-irradiation times (in seconds) are shown inset.

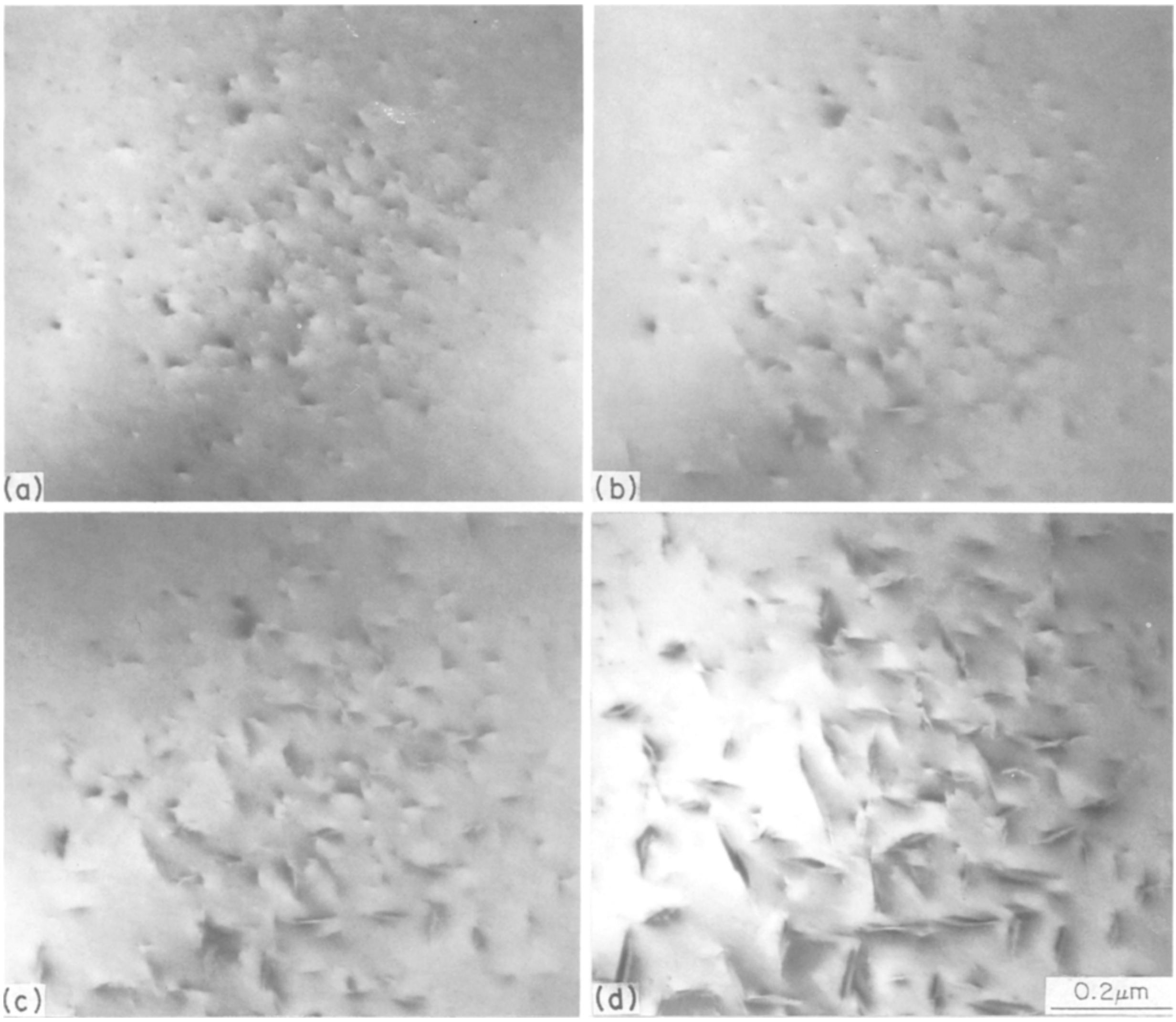

Figure 6 Effect of re-irradiation at 400° C with a high electron flux ($2 \times 10^{24}\,m^{-2}\,sec^{-1}$) on silicon previously irradiated at 100° C. The re-irradiation times are (a) 36 sec, (b) 75 sec, (c) 105 sec, (d) 162 sec. The defects produced by the first irradiation are seen to grow.

of the small loops remains unchanged. It is found that the incubation time also corresponds to an accumulated electron dose of about $2 \times 10^{26}\,m^{-2}$. It must be noted here that the loops which vanish during the re-irradiation are those produced in the bulk by the first irradiation. The present work also indicated that the re-irradiation could not cause the disappearance of the loops near the specimen top surface, which were formed after a long irradiation period in both Cz and Fz silicon specimens.

Re-irradiation of Cz silicon at 400° C after the first irradiation at temperatures below 200° C was also performed. When the flux used was not too strong, as reported by Furuno *et al.* [6], the small loops produced by the first irradiation disappeared and at the same time larger loops appeared. However, when a high electron flux above $10^{24}\,m^{-2}\,sec^{-1}$ was used, the effect of the re-irradiation was quite different. In Fig. 6 is shown an example. The small defects produced by the first irradiation at 100° C are found to continue to grow under the second irradiation at 400° C.

3.4. Determination of the nature of the small loops

To investigate the nature of the small loops formed at temperatures below 200° C, the $2\frac{1}{2}$D method [11] was employed: A small number of fine gold particles were vacuum evaporated onto one surface of the specimen as a reference strain free material and then a pair of dark-field under and overfocussed micrographs were taken. From the relative image shifts of the small loops with respect to the gold particles, it was found that the small loops were of interstitial type.

The type of the loops was also confirmed by the inside–outside contrast method [12, 13] after growing them by the second irradiation at 400° C as shown in Fig. 6. The habit planes of the loops were first determined to be $\{1\,1\,3\}$ planes by using the micrographs taken from the $\langle 2\,1\,1 \rangle$ pole like Fig. 7. The Burgers vector, $\boldsymbol{b}$, of the $\{1\,1\,3\}$ loops was found to be along the $\langle 1\,1\,6 \rangle$ directions, as reported before for those formed at high temperatures [7], from the fact that the images of the loops vanished when $\boldsymbol{g} = 2\bar{2}0$ or $\boldsymbol{g} = 51\bar{1}$ was excited. In Figs 8a and b a pair of micrographs for the inside–outside contrast analysis taken near the [1 0 0] pole in (a) $g = 0\bar{2}0$ and (b) $g = 020$, respectively, is shown. The deviation parameters from the exact Bragg condition, s, are positive in both (a) and (b). The loops lying on $(1\,\bar{3}\,1)$, $(1\,\bar{3}\,\bar{1})$, $(1\,3\,\bar{1})$ and $(1\,3\,1)$ planes (see for instance, A, B, C and

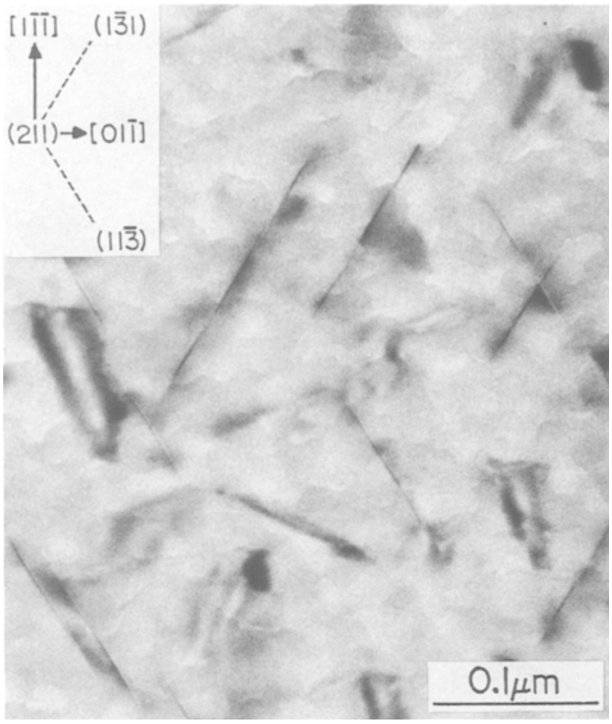

Figure 7 Image along the [2 1 1] direction showing loops lying on various {3 1 1} planes. The loops on the (1 1 $\bar{3}$) and (1 $\bar{3}$ 1) planes are parallel with the incident electron beam and are seen as lines.

D, respectively) are clearly visible among other loops lying on other planes and are analysed. Table I shows the result of the analysis. The normal of the loop, ***n***, shown in the second column is determined in the sense that it points upwards in the electron microscope, i.e. making an obtuse angle with the direction of viewing, which is the [1 0 0] direction in the present case. In the third and fourth columns are shown the sign of the quantity (***g*** · ***b***)s for each loop in (a) and (b) of Fig. 8, determined as positive or negative by observing whether the loop gives inside contrast or outside contrast, respectively. Since ***g*** and the sign of *s* are known, the sense of ***b*** can be determined, which is shown in the fifth column. Finally, the sign of ***n*** · ***b*** for each loop

TABLE I Analaysis of the nature of the loops originated during irradiation at 100° C and developed by the second irradiation at 400° C

Loop	***n***	(***g*** · ***b***)s (a) $g = 0\bar{2}0$	(***g*** · ***b***)s (b) $g = 020$	Direction of ***b***	***n*** · ***b***
A	$\bar{1}3\bar{1}$	>0	<0	$1\bar{6}1$	<0
B	$\bar{1}31$	>0	<0	$1\bar{6}\bar{1}$	<0
C	$\bar{1}\bar{3}1$	<0	>0	$16\bar{1}$	<0
D	$\bar{1}\bar{3}\bar{1}$	<0	>0	161	<0

obtained by multiplying ***n*** by ***b*** is shown in the last column and all of them are negative. This shows that the loops are formed by shifting the bottom surface of the loop planes against the top surface, i.e. they are of interstitial type [13].

The loops lying on (1 1 3), ($\bar{1}$ 1 3), (1 $\bar{1}$ 3) and (1 1 $\bar{3}$) and (3 1 1), ($\bar{3}$ 1 1), (3 $\bar{1}$ 1) and (3 1 $\bar{1}$) planes which are almost invisible in Fig. 8 were investigated with a number of other reflections, and they were also found to be of interstitial type.

3.5. Re-irradiation from the reverse side of the specimen

To clarify why the secondary defects are formed only near the specimen top surface, the specimens previously irradiated at 200° C were turned upside down and then irradiated again at the same temperature. The *in situ* observation sequence is shown in Fig. 9. The loops seen in Fig. 9a are those produced by the first irradiation, which are situated near the bottom surface of the specimen during the re-irradiation. As the irradiation proceeds, these defects vanish and at the same time new defects appear. It was found that this also occurred in the case of Fz silicon. One can easily notice that all the new defects show the opposite black–white contrast from those situated near the bottom surface. This fact indicates that the new defects are formed near the top surface, which is confirmed by stereomicroscopy.

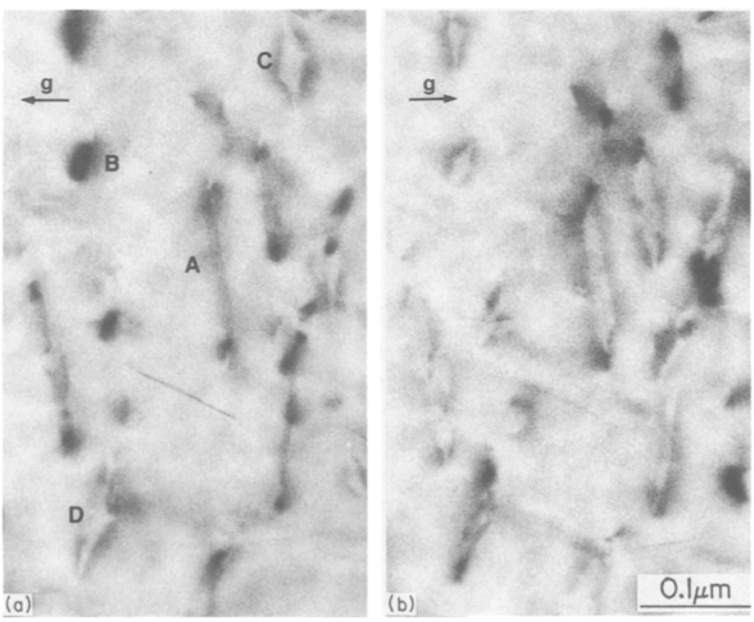

Figure 8 Micrographs taken in (a) $g = 0\bar{2}0$ and (b) $g = 020$ reflections, showing the inside–outside contrast of the loops in silicon.

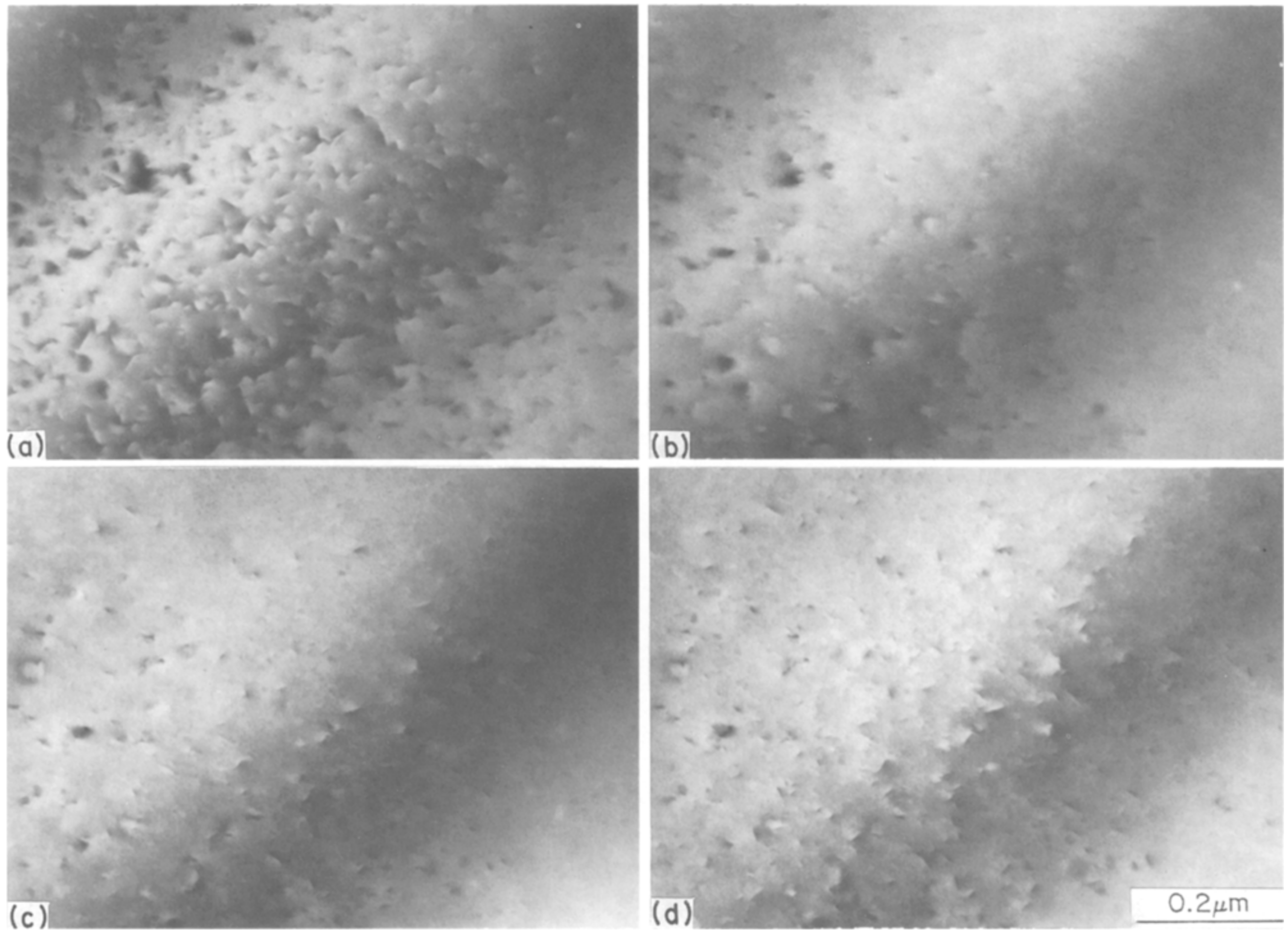

Figure 9 Effect of re-irradiation on a silicon specimen previously irradiated at 200° C after turning it upside down. The re-irradiation times are (a) 55 sec, (b) 315 sec, (c) 574 sec, (d) 665 sec.

4. Discussion

4.1. Two processes of formation of secondary defects

In the case of Fz silicon, the electron irradiation induced secondary defects were formed only in the localized region near the top surface of the specimen after a certain incubation period [7–9]. In the case of Cz silicon, however, the secondary defects were observed to form in the bulk of the specimen immediately after the irradiation started [9]. It was also found in the present work that the formation of the secondary defects near the top surface of the Cz silicon specimens took place after a prolonged irradiation, as in the case of Fz silicon.

From these results, it is clear that the secondary defects, all of which are found to be interstitial type dislocation loops, can be divided into two groups, according to their nucleation characteristics; (1) the interstitial loops formed in the bulk of Cz silicon specimen, nucleation of which is closely related to the large number of oxygen atoms contained in the crystal, and (2) the interstitial loops formed near the electron incident surface of the specimen after a long irradiation in both Cz and Fz silicon specimens, nucleation of which is stimulated by an effect from the surface.

As mentioned in the first section, Furuno *et al.* [6] performed a series of re-irradiation experiments and concluded that the small defects formed below 200° C were of vacancy type. In the present work, the formation of the small defects after the disappearance of larger interstitial loops was also observed. However, these defects were found to have a different distribution in the specimen from that of the larger interstitial loops, and to be interstitial in nature by using more direct determination methods of the $2\frac{1}{2}$D method and the inside–outside contrast method. In addition, it is clear from Fig. 5 that the formation of the small defects is independent of the shrinkage of the pre-existing interstitial loops. While the shrinkage speed of the interstitial loops depends on the re-irradiation temperature strongly, the formation of the small defects only depends on the total electron dose. Therefore, it is concluded that the shrinkage of the interstitial loops is a result of the increasing of the vacancy concentration in the bulk of the specimen but the formation of the small loops is due to some effect from the specimen surface during the irradiation. In other words, the larger loops belong to the first group of the secondary defects and the small loops belong to the second group of the secondary defects; two of them are independent of each other.

Detailed possible formation mechanisms of these defects will be considered in the following.

4.2. The formation and growth of the interstitial loops in the bulk of Cz silicon specimens

The formation and development of the interstitial loops in the bulk of Cz silicon specimens show very clear dependence not only on the irradiation temperature but also on the electron flux density and the specimen thickness.

At temperatures below 250° C, unless the specimen is thicker than 500 nm, no interstitial loops were formed in the bulk of Cz silicon specimen. In addition, the existing loops formed by the irradiation at 400° C shrunk and disappeared when they were re-irradiated at temperatures below 250° C. These results clearly suggest that at low temperatures the concentration of the interstitials in the bulk of the specimen is considerably lower than that of the vacancies. Such a situation is likely to occur because the mobility of the vacancies is much smaller than that of the interstitials especially at low temperatures. Therefore, the vacancies are accumulated in the crystal unless annihilated with interstitials, while the remnant interstitials migrate fast to the sinks such as the specimen surfaces and to the periphery of the irradiation area at low temperatures.

In the cases of many metals and alloys, the more intensive the electron flux is, the higher the density of the secondary defects and the more rapid their growth. In the present case of Cz silicon, however, when the electron flux is higher than $10^{24}\,m^{-2}\,sec^{-1}$, the interstitial loops are not formed in the bulk of the specimen, as shown in Fig. 1. Only in the thick areas of the specimen as in Fig. 2, are the loops formed in the beginning of the irradiation. However, these loops, especially those in the central area, start to shrink gradually after 50 sec and finally disappear. This implies that the concentration of the interstitials and vacancies has not reached a steady state in the specimen at least until this time. This can be explained if some vacancy clusters are formed gradually, on a submicroscopic scale, with the irradiation. For instance, divacancies, which are known to be the major defects in electron, neutron or ion irradiated silicon at room temperature, should be considered. Svensson *et al.* [14] investigated the generation of divacancies in tin-doped silicon samples during 2 MeV electron irradiation. They found that the vacancy–vacancy pairing is the dominating mechanism for formation of divacancies at room temperature. Since the binding energy of divacancy, which is estimated to be at least 1.6 eV [15], is large, and also in the present case the production rate of vacancies is very high, it can be expected that the concentration of divacancies increases as the irradiation proceeds even at higher temperatures of the present case. As a result of the formation of the divacancies, the sinks for interstitials are increased so that the concentration of them in the specimen decreases. For this reason the loops formed absorb more vacancies than interstitials, and shrink. There is of course a probability of formation of other larger clusters of vacancies.

4.3. The formation of defects near the specimen top surface

Irrespective of Cz and Fz silicon, the interstitial loops were formed near the top surface of the specimen when the total electron dose reached about $2 \times 10^{26}\,m^{-2}$. The results shown in Figs 1, 2 and 5 clearly indicate that there is a large difference in the interstitial concentration between the bulk and the region near the specimen top surface during irradiation. While the concentration of the interstitials in the bulk was shown to be low or decreasing, new interstitial loops appeared in the region near the top surface as the irradiation proceeded. This is possibly due to a certain effect from the surface, which is known to be covered by a thin oxide layer. It was first proposed by Hu [16] that the oxidation of silicon generates excess self-interstitials. If the oxygen atoms are shot in from the surface oxide layer by the irradiation, they will be precipitated to eject silicon interstitials. This could be the reason for the formation of the interstitial defects near the top surface.

The result shown by Fig. 9 seems to support this model. If the formation of the defects only originates from the nucleation process due to some impurities from the surface, it cannot be explained why the defects previously formed disappear when the specimen is turned upside down.

Investigations on the silicon specimen surface before and after irradiation are necessary to make the mechanism clearer.

5. Conclusions

The formation of electron irradiation induced secondary defects in Cz silicon crystals was studied mainly by using a ultra-high voltage electron microscope. A small number of Fz silicon specimens were also examined for comparison. The results are summarized as follows.

(1) The small defects formed at low temperatures are, as well as the larger defects formed at high temperatures, of interstitial type.

(2) During the irradiation supersaturation of interstitials occurs in the region near the specimen top surface, i.e., the electron incident surface. This supersaturation may be caused by precipitation of the oxygen atoms knocked in from the surface oxide layer. The formation of the interstitial defects near the top surface is most likely due to the local interstitial supersaturation.

(3) The mechanisms of the formation of the electron irradiation induced secondary defects are divided into two categories. One is that the defects are formed in the bulk of Cz silicon by the nucleation process due to the oxygen atoms contained in the crystal and the other is the defect formation in the region near the top surfaces of both Cz and Fz silicon specimens, arising from the local supersaturation of the interstitials.

(4) The results obtained in the present study strongly suggest that some invisible vacancy clusters are formed during the irradiation, especially under a high electron flux or at low temperatures. The most probable clusters are the divacancies.

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