SOLIDS

MICRODEFECTS FORMATION IN DISLOCATION-FREE FLOAT-ZONE AND CZOCHRALSKI SILICON SINGLE CRYSTALS

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Basing upon complex researches of monocrystals FZ-Si, a mechanism of microdefect formation is confirmed. It is established that the formation of microdefects happens by two mechanisms: vacancy and interstitial ones. The comparison of the data on CZ-Si and FZ-Si shows that this mechanism can be applied to CZ-Si with allowance for a modification of growth conditions and is significant for a larger content of impurities.

Structural microdefects formed during a cooling of float-zone (FZ) and Crochralski (CZ) crystals after their growth may include the agglomerates of point defects and impurities. These structural defects can detrimentally affect the reliability of semiconducting devices and their performance. Since only high-purity silicon can be used in modern electronic industry, knowledge of defect formation processes in a semiconducting material is necessary.

In this paper, we inform about the mechanism of formation of microdefects in monocrystals FZ-Si recently determined by us and make attempt to apply it to monocrystals CZ-Si.

1. Microdefect Formation in FZ-Si

By summarizing all experimental results about the physical nature of microdefects in dislocation-free monocrystals FZ-Si with a diameter of 30 mm, we present the following classification [1]:

1) A-microdefects are interstitial dislocation loops with sizes of $1 \div 50$ μm with the Burgers vector $\bar{b} = 1/2$ [110], which are in planes {111} and {110}.

2) B-microdefects are point defects agglomerates of the interstitial type with sizes of $20 \div 50$ nm. A part of them is in planes {100}. By TEM, they are represented as rectangles and rhombs in plane {111} with parties on directions [110] and [100], respectively.

3) D-microdefects of the interstitial type are agglomerates of point defects with sizes of $4 \div 10$ nm. Considering them as small dislocation loops, it is possible to conclude that they can be in planes {100}, {110}, {$\bar{1}$11} and have Burgers vectors $\bar{b} = 1/2$ [100] and $\bar{b} = 1/2$ [110].

4) C-microdefects are completely identical to D-microdefects on TEM-images, as well as the sign of deformation of the crystalline lattice and their sizes.

5) It is suggested [2] that D-microdefects are uniformly distributed B-microdefects.

In crystals which were obtained at high growth rates (more than 6 mm/min), microdefects of the vacancy type are formed simultaneously with microdefects of the interstitial type in the same regions of a crystal.

However, using X-ray topography followed by decoration with copper, it was suggested [3] that D-microdefects have a vacancy character in Czochralski-

Microdefects classification in FZ-Si

Type	Physical nature	Size, nm	V, mm/min	Distribution in plane (112)	N, cm^{-3}	Refs.
A	Interstitial dislocation loops	up to 50000	$1 \div 3.5$	Stratified	$\sim 10^6$	[2, 4÷7]
B	Interstitial agglomerates of point defects	$20 \div 50$	≤ 4.5	Stratified	$\sim 10^7$	[2, 4÷7]
C	Interstitial agglomerates of point defects	$4 \div 10$	> 4.5	Uniform	$\sim 10^{13}$	[1,2,6,8,9]
D	Interstitial aglomerates of point defects	$4 \div 10$	> 4.5	Uniform	$\sim 10^{13}$	[1,2,6,8,9]
$I + V$	Interstitial + vacancy	$4 \div 12$	> 6	Uniform	$\sim 10^{13}$	[2,6,8]

grown silicon. The results [3] cannot be represented as 'incorrect". Since vacancy microdefects and interstitials microdefects coexist, the vacancy microdefects detected in [3] are not D-microdefects according to the classification given in [1]. The difference between the classifications [1] and [3] is not basic, but they can cause disagreement in the determination of microdefects types. Here, we use the classification of [1]. In Table, the literary information about a microdefects classification in FZ-Si is shown.

Defects which are identical to uniformly distributed D-defects in FZ-Si are not observed in CZ-Si. Thus, we think that, similarly to defects in FZ-Si, defects in CZ-Si were called 'D-defects" [3]. Such defects exist in the areas of a crystal where the coexistence of interstitial and vacancy defects are revealed. In Table, these areas are marked as '$I + V$". In [3], CZ-Si was studied, where the revealing of interstitial D-microdefects is very difficult in the comparison with FZ-Si. However, we think that the processes of defects formation in both these types of crystals are identical [10].

Various theoretical models were suggested to explain the regularities of microdefects formation in silicon. Main problems were the dominant type of point defects in a crystal, their concentration, and mutual interaction. In some models [5, 7, 11, 12], it was assumed that the dominating type of point defects in crystals are interstitical atoms of silicon. In other models [13 ⁻ 16], it was supposed that the dominating type of defects is vacancies. In contrast, simultaneous independent coexistence of both main types of point defects at high temperatures was suggested in [17, 18]. None of these models could explain experimental results, which were obtained later with the help of TEM, as was shown in [19].

According to the commonly accepted Voronkov theory [20 ⁻ 22], the recombination rate between isolated vacancy and interstitial defects is high and the further diffusivity of interstitials is higher than the diffusivity of vacancies near the melting point. Finally, the concentrations of vacancies are higher than the concentrations of interstitials at the melting point, where both the concentartions are in the thermal equilibrium. Only the microdefects of the interstitial type (A- and B-microdefects if the concentration of interstitial atoms is higher than the concentration of vacancies) or only the vacancy type (D-microdefects if the concentration of vacancies is higher than that of interstitial atoms) are formed in the crystal. According to [21], the type of dominating point defects depends on the parameter V/G (V ⁻ growth rate of a crystal, G ⁻ axial temperature gradient): if $V/G < C_{crit}$, then interstitial atoms of silicon dominate in the crystal, but if $V/G > C_{crit}$, then the vacancies dominate.

However, these results, whose theoretical interpretation was not mainly changed since 1982,

cannot explain some latest experimental results [2, 6, 8, 9, 23 ⁻ 28]. Furthermore, the Voronkov theory does not take into account the influence of carbon on the process of microdefects formation. The participation of carbon during defect formation results in a conclusion about a heterogeneous character of nucleation of microdefects [26, 27]. In TEM studies [8], it was established that the equilibrium concentrations of vacancies and self-interstitials are approximately identical at temperatures close to the silicon melting point. Furthermore, the Voronkov theory does not explain the nature of 'defect-free" areas in silicon. In a 'defect-free" area, microdefects of a crystalline lattice of both types were observed in [8].

1.1. Experiments and Discussion

With the purpose of clarification of disputable positions, we conducted the researches of monocrystals FZ-Si with a diameter of 30 mm. Non-doped monocrystals of high resistivity (2200 ⁻ 4000 $\Omega \cdot$ cm) from n-type silicon were grown by the float zone technique in vacuum. The number of passes of a melting zone varied from 2 up to 10. The concentrations of oxygen and carbon determined by IR-absorption were less than $5 \cdot 10^{15}$, $\ddagger^{-3}$. The crystals were obtained with various growth rates (1 ⁻ 9 mm/min) and were subjected to various kinds of thermal processings. The methods of optical microscopy X-ray topography and TEM were used. The technique of our experiment is explicitly shown in [29 ⁻ 32]. Temperatures of formation of various microdefects types are experimentally determined [29, 30], the and transformational mechanism of interstitials microdefects is established [31]. It is established that, in crystals obtained at $V > 6$ mm/min, interstitial microdefects occur with vacancy microdefects whose concentration is increased with the crystal growth rate. It is established by TEM that the equilibrium concentrations of vacancies and interstitial atoms are approximately identical near the smelting point [10]. It is established that only interstitial microdefects will be formed for $V/G < C_{crit}$ (A- and B-microdefects reveal the stratified distribution, and D-microdefects do the uniform distribution). With the help of TEM, it is established that, in cristals obtained at $V > 6$ mm/ min. i.e. for $V/G > C_{crit}$, vacancy microdefects occur with interstitial microdefects. The ratio of vacancy and interstitial microdefects in samples obtained at $V = 7.5$ mm/min is approximately equal to 1:4. In crystals obtained at $V = 9$ mm/min, interstitial and vacancy defects coexist approximately with identical concentrations. Thus, the critical growth rate of a crystal, for which vacancy microdefects occur, is in the interval $6 < V < 6.5$ mm/min [32].

Furthermore, our previous researches [2, 9] by TEM revealed two types of images of *D*-microdefects: the agglomerates of atoms with crystalline and amorphous structures (Fig. 1). In [9], it was shown that one of such images givens microprecipitates of amorphous phases of SiO_2. We suppose that the crystalline structure gives microprecipitates SiC [33]. Both types of *D*-microdefects are defects of the interstitial type.
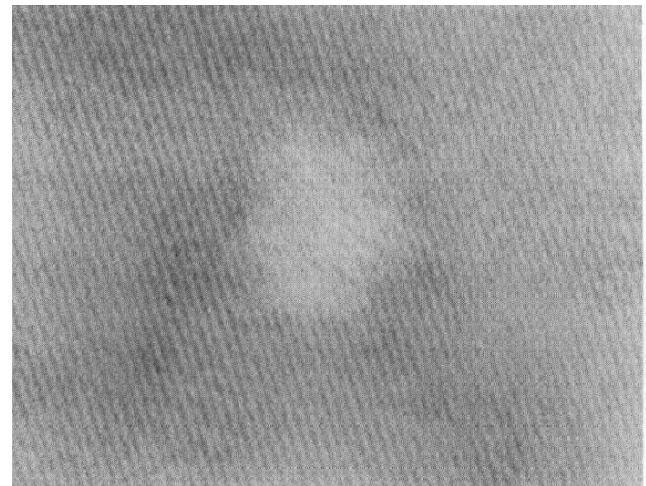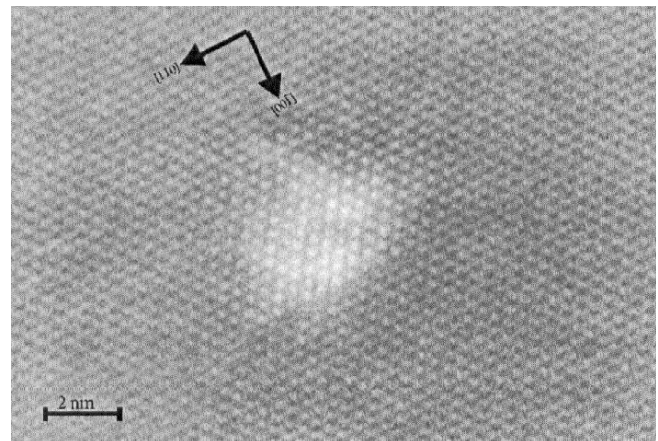
All these results show that most microdefects have two paths of nucleation. The impurities of carbon and oxygen facilitate the process of nucleation of microdefects.

In [21] it is suggested that the main role on the initial stage of disintegration of the oversaturated solid solution of point defects is played by the process of recombination of vacancies and self-interstitials.

However, it was shown [34 ¯ 36] that point defects in silicon have an extended character at very high temperatures, i.e. one atom (or one vacancy) 'is extended" over several nuclear volumes. According to this model, recombination happens only with the help of simultaneous compression of these defects in a neighbourhood of one nuclear volume. In view of this, it was shown [37, 38] that the recombination of point defects on the initial stage of their interaction at a high temperatures is hindered by an activation barrier, which includes the enthalpy and entropy components. The main contribution is given by the entropy component, whose value reaches 11 ¯ 11.5 k [38]. The barrier is decreased if temperature is lowered, and it disappears at low temperatures. Thus, the disintegration of the oversaturated solid solution of native point defects (formation of microdefects) proceeds simultaneously by two mechanisms: vacancy and interstitial ones [10, 32].

Theoretically, only the vacancy aggregation and joint vacancy-impurities one are possible for the vacancy mechanism [21]. For FZ-Si crystals, the vacancy-impurities aggregation (vacancy + atoms of oxygen) begins earlier than the vacancy aggregation. The absorption of vacancies and impurities by growing microdefects results in a decreased concentration of vacancies as compared with the concentration of oxygen. As a result, precipitates begin to absorb oxygen without participation of vacancies, their sizes are increased, and then the type of a strain around them varies from the vacancy to interstitial one. The boundary of the full transition can be determined by the ratio V/G. Thus, the parameter C_{crit} of the theory [21] does not describe the condition for growth mode change (interstitial or vacancy one). This parameter describes the conditions of vanishing (emerging) of microdefects of the vacancy type due to diffusion and the interaction of point defects during cooling of a crystal.

For the interstitial mechanism, the catalytic role is played by carbon atoms. The growth of interstitial microdefects results in a significant lowering of the
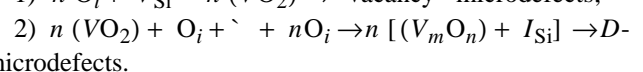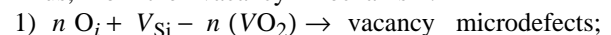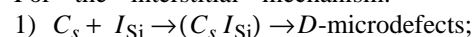

Fig. 1. TEM-image of *D*-microdefects: crystalline phase (*a*), amorphous phase (*b*)

concentration of self-interstitials. This creates conditions for the precipitation of an impurity. In this case, the formation of particles of the impurity phase is accompanied by generation of self-interstitial atoms at internal positions of the lattice. As a result, two types of interstitial microdefects will be formed: interstitial congestions (drains for interstitial atoms of silicon) and impurity precipitates (sources of these atoms). Both mechanisms (vacancy and interstitial ones) result in the formation of small interstitial congestions, i.e., *D*-microdefects. These congestions are uniformly distributed small *B*-microdefects.

Thus, for the vacancy mechanism:

1) $n\,O_i + V_{Si} - n\,(VO_2) \to$ vacancy microdefects;

2) $n\,(VO_2) + O_i + \,` + nO_i \to n\,[(V_mO_n) + I_{Si}] \to D$-microdefects.

For the interstitial mechanism:
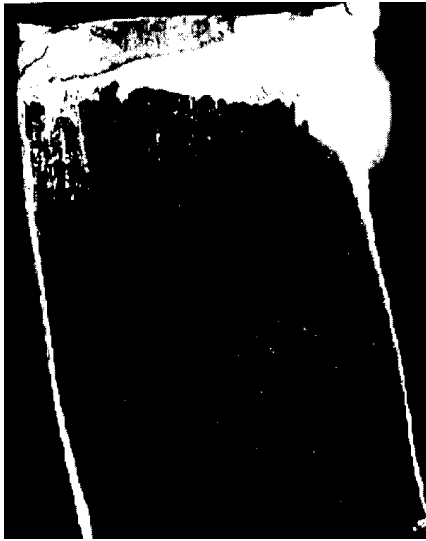
1) $C_s + I_{Si} \to (C_s I_{Si}) \to D$-microdefects;

Fig. 2. Distribution of microdefects of various types at $V = 6$ mm/min

2) $(C_s I_{Si}) + O_i \rightarrow n [(C_s I_{Si}) + O_i] \rightarrow B$-microdefects;

3) B-microdefects $+ I_{Si} \rightarrow A$-microdefects.

Basing on our experimental data, the following mechanism of transformation of interstitial microdefects was offered [31]. Agglomerates in planes {100} generate self-interstitials. The agglomerates of interstitial atoms promote the formation and growth of dislocation loops in planes {111}. Thus, D-microdefects which are the predecessors of B-microdefects can exist in two types: as flat agglomerates of point defects in planes {100} and as small dislocation loops in planes {111}. The further growth of agglomerates in planes {100} results in the generation of dislocation loops in planes {110}. Such a process happens at the expense of the mechanism of prismatic extrusion. As a result, A-microdefects will be formed as dislocation loops in planes {111} and {110} with the Burgers vector $\bar{b} = 1/2 [110]$. Therefore, the transformation of interstitial microdefects proceeds according to the following scheme: D-microdefects $\rightarrow$ B-microdefects $\rightarrow$ A-microdefects. During the transformation of interstitial microdefects with a lowering of growth rate, the size of defects is increased and their concentration decreases.

2. Microdefects in CZ-Si

Because of a modification of the temperature conditions of growth, a greater variety of defect formation paths is observed in CZ-Si [39]. It was established that A- and B-microdefects in CZ-Si are identical to the same defects in FZ-Si [40]. Microdefects in crystals obtained at high growth rates, existing at concentrations of $10^5 - 10^7 \, cm^{-3}$, were detected and were called A'-

microdefects [41]. It was assumed [22, 41] that these defects are completely similar to 'D-microdefects" that were detected in [3], i.e. have the vacancy character. The same defects were determined by other methods and named 'flow-patterns defects (FPDs)" [42], defects observed by IR laser scattering tomography (LSTDs) [43], and others. Recently grown-in LSTDs were observed by TEM [44, 45], and their form was determined as octahedron. Basing on a weaker oxygen signal from them in comparison with oxide precipitate, it was made the conclusion to consider these defects as voids [44, 45], i.e. vacancy clusters with oxygen impurity [22, 46]. Here, we call these defects by their initial name, i.e. A'-microdefects. According to [21], their formation is observed in the 'vacancy" mode of crystal growth, i.e. in the 'vacancy" area of a crystal $(V/G > C_{crit})$. Furthermore, it was revealed that, between the 'vacancy" and 'interstitial" areas, a 'defect-free" area exists, where there is no process of oxygen precipitation [47]. On a rim of the area of A'-microdefects, 'the zone of anomalous oxygen precipitation" is observed as an oxidation-incduced stacking-fault ring (OSF-ring), but there are presumably vacancy defects inside it [48, 49].

But there are also other publications, where the results do not correlate with results of the marked publications. In [50], tiny defects by a size ca 10 nm with high concentration $(10^{13} - 10^{14} \, cm^{-3})$ were observed by TEM. Size, concentration and image of these defects are completely identical to those of D-microdefects of the interstitial type, which were observed in FZ-Si [2, 6]. Very interesting results were obtained in [23]. It was established [23] that, in the 'defect-free" area, there are two types of defects with different signs of a strain. It was assumed [23] that A'-microdefects are microparticles SiO_x with specific volume large than that in Si. These results are identical to the results obtained by us in FZ-Si and concerning the coexistence of microdefects of the vacancy and interstitial types.

The distribution images of microdefects in FZ-Si crystal, which was obtained by us at $V = 6$ mm/min after quenching, are shown in Fig. 2. In these crystals, the 'defect-free" area was formed. The 'defect-free" area contains very small defects of the interstitial and vacancy types, as was revealed by TEM [30]. The size of these defects was $3 - 7$ nm and their density was $\approx 3.5 \cdot 10^4 \, cm^{-2}$, whereas the same defects have a double size more and density three times less $(\approx 1.2 \cdot 10^4 \, cm^{-2})$ in the area with D-microdefects than in 'defect-free" areas. We may consider these defects as the complexes of vacancies and interstitial atoms of oxygen O_i. These complexes are the predecessors of D-microdefects. It is possible that they are $[V O_2]$ complexes that were detected in silicon in radiation experiments [51].
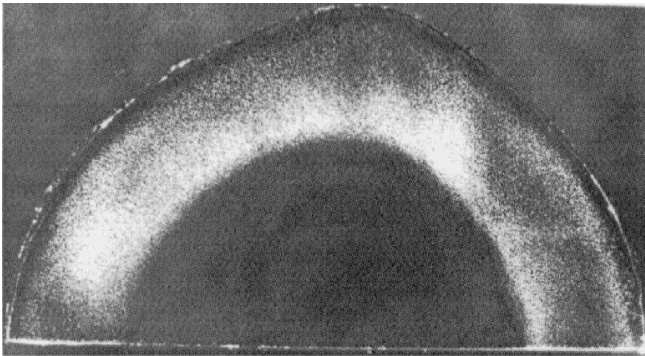
Fig. 3. 'Ring" of *D*-microdefects in FZ-Si at *V* = 7.5 mm/min

Thus, some question about the nature of an OSF-ring and area with *A '*-defects requires the explanation. It is established that there are aggregates of oxygen in an OSF-ring [52]. It was assumed [22, 53] that vacancy microdefects are responsible for the formation of OSF-rings. In [27], it is shown that there are two paths of nucleation of OSF-rings: by formation of point-defect clusters (interstitial atoms and unoxygen impurities) and oxygen precipitation.

It was established [39, 49] that if the growth rate is lowered, then the ring diameter shrinks. Similar rings were observed in FZ-Si for the first time in [6]. We performed detailed TEM-researches of such crystals (Fig. 3). It was established that the ring is a congestion of *D*-microdefects of the interstitial type which are formed by the vacancy and interstitial mechanisms. In the area inside a ring, coexisting microdefects of the vacancy and interstitial types are observed by TEM. In FZ-Si as well as in CZ-Si, the diameter of a ring shrinks with a lowering of the growth rate.

Recently, LSTDs were observed by using the method of synchrotron radiation topography (SRT) [24]. It was assumed that these defects are of the interstitial type. Clusters of the interstitial type in CZ-Si were observed by TEM [54]. It was shown that the sign of a strain of LSTDs, i.e.. *A '*-defects, has interstitial character [25, 26]. Therefore, it was concluded [25] that two types of defects were involved in the grown-in defect formation process: the most important role is played by agglomerations of self-interstitial Si atoms + 'unknown impurity". It was assumed [25] that this impurity can be carbon which takes active participation in the void formation [55].

Thus, both groups of data on monocrystals CZ-Si show that, in these crystals, the microdefect formation happens by two mechanisms as well as in FZ-Si. In CZ-Si as well as in FZ-Si, the simultaneous coexistence of microdefects of the vacancy and interstitial types is observed. They can be found in 'areas with *A '*-defects". Therefore, some authors observed vacancy

defects in this area, whereas others found interstitial defects in the same area. So, it is possible to assume that the process of formation of microdefects in CZ-Si is similar to the same process in FZ-Si. Therefore, the model of microdefect formation for FZ-Si can be applied to CZ-Si.

Conclusion

To conclude, we note that the experimental data obtained for FZ-Si correlate well with the experimental data obtained by different authors with crystals CZ-Si. Therefore, the physical model that describes the processes of nucleation and growth of microdefects in FZ-Si can be applied to CZ-Si with allowance for a modification of growth conditions and for a larger content of impurities.

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