

Radiative properties of dislocations generated around oxygen precipitates in Si

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Generation of dislocations during the growth of oxygen precipitates has been used as an alternative way of introduction of dislocation-related luminescence centers. For this purpose a multistep annealing of Cz Si samples with different initial concentrations of oxygen has been carried out. The analysis of defect density and structure was performed by optical microscopy (OM) and transmission electron microscopy (TEM). The dislocation-related luminescence (DRL) appeared only after a growth stage, while its intensity strongly depended on the duration of the preliminary nucleation treatment. The duration of growth annealing had a strong influence on the spectral distribution of the DRL intensity. No correlation has been found between a particular defect density, defined by TEM, and the shape of luminescence bands. Therefore, it was concluded that the cause of the gradual DRL transformation is redistribution of oxygen, collected near dislocations.

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1 Introduction

The study of DRL has two objectives. First, it is important as a practical matter for developing all-silicon light-emitting diodes (LED). On the other hand, there are some interesting physical aspects of radiative recombination at deep levels in semiconductors with an indirect energy gap. The successful fabrication of LED based on dislocations in silicon [1] requires development of a more technological way for dislocation introduction. In this context, the generation of dislocations during the growth of oxygen precipitates can be considered as an alternative way. The precipitation practice is already widely used for internal gettering, therefore it is approved in technological processes.

The annealing of Cz Si samples at temperatures far below the melting point causes oxygen to diffuse and agglomerate in the form of amorphous SiO₂ precipitates due to supersaturation. The resulted particles would be expected to have various sizes and shapes. Very often, they are the origin of other defects, such as dislocation loops, dislocation dipoles and stacking faults. This process is controlled by the diffusion coefficient, therefore at lower temperatures the precipitate sizes are very small [2, 3]. The low-temperature annealing is usually considered as a nucleation treatment. The nucleation stage serves for preparation of a given site density [4], while the growth stage defines the final density and sizes of precipitates, the dislocation morphology and the state of oxygen atmosphere near to and at dislocations.

The most promising band in DRL, D1 with energy position about 0.8 eV is very sensitive to oxygen contamination [5], and to dislocation morphology [6]. Depending on thermal treatment, its maximum

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varies from 790 to 850 meV. The spectral distribution of intensity in the D1 band is determined by the number and the state of oxygen atoms or small clusters near to the dislocations [5]. In spite of extensive studies the microscopic structure of the centers responsible for D1/D2 bands is not clear yet. Nevertheless, it is known that they are point defects with tetrahedral symmetry and are situated near to or at a core of dislocations [7]. The relative intensity of the D1 band rapidly increases with dislocation density. This implies that generation of the D1 centers is enhanced by interaction between dislocations [6]. Several bands can be found in the range of the D1 band, the intensity and position of which depend on the thermal treatment history. Different models have been suggested for the origin of these bands. The model of donor–acceptor pair recombination enabled a fit of the spectral distribution of PL intensity at the low-energy shoulder of D1 band in FZ silicon with reasonable accuracy [8]. On the other hand, oxygen rich Cz Si samples very often demonstrate the high-energy band of the D1 family with energy position varying in the interval from 0.807 to 0.850 eV. The dependence of the position of this band on the excitation power and oxygen concentration led to the tentative assumption of the same donor–acceptor recombination mechanism as for the low-energy band, but with different types of thermal donors [5]. Another model for explanation of the high-energy band has been suggested in Refs. [9–11], where recombination of carriers captured at a strain-induced potential around oxygen precipitates has been considered.

Local density and morphology of dislocations, created near precipitates, strongly depends on their types, sizes and growth kinetics. Therefore, we can expect that the concentration and state of D1 centers generated near precipitates would strongly depend on the parameters of thermal treatment during the nucleation and growth stages, and on the initial oxygen concentration ($[O_i]$).

The objective of this study is to define the relation between the conditions of precipitation procedure and generation and properties of DRL centers. This will be done on the basis of comparative investigation of defect structure and DRL.

2 Experimental

Due to the fact that oxygen plays a decisive role in process of formation of structural defects, three types of Cz Si samples have been used in experiments with oxygen concentrations $[O_i]$ of $6 \times 10^{17} \text{ cm}^{-3}$, $8 \times 10^{17} \text{ cm}^{-3}$ and $\geq 10 \times 10^{17} \text{ cm}^{-3}$, called hereafter low, medium and high concentration. The $[O_i]$ were determined by a standard FTIR technique at room temperature. The accuracy of determination of $[O_i]$ was at least $5 \times 10^{16} \text{ cm}^{-3}$, which is much less than the difference between the samples. Before a particular thermal treatment all samples were annealed at 1000 °C for 15 min to have equal starting conditions. Multistep annealing of samples was performed for generation of precipitates. This included the nucleation treatment at 650 °C with steps of 6, 8, 16 and 32 h, followed by a stabilization heat treatment at 800 °C for 4 h, and growth annealing at 1000 °C.

Some samples were treated using a rapid thermal annealing (RTA) at 1250 °C in an argon atmosphere as a starting treatment. This procedure is effective for creation of a so-called “magic denuded zone”, therefore the samples are called hereafter MDZ samples. In this case, the produced nuclei density is independent of oxygen content. The annealing at 800 °C for 4 h serves for MDZ samples as the nucleation step. The use of an RTA treatment is based on the influence of vacancies on a decay rate of oxygen solid solution. When the concentration of vacancies exceeds the critical level of $1 \times 10^{12} \text{ cm}^{-3}$, the decay rate increases rapidly [12]. In addition, RTA treatment allows formation of a required profile of vacancy distribution in depth of sample and control of the decay rate of oxygen solid solution.

The density and structure of defects were carefully analyzed by chemical etching and TEM. Usually, many foils were prepared from each sample and analyzed to obtain reliable data on a particular defect concentration.

The PL spectra were recorded using a conventional lock-in technique with a cooled Ge detector. The samples were placed to a variable temperature He cryostat. PL was excited by a GaAs laser with P_{exc} of about 1 W/cm^2 and $\lambda = 920 \text{ nm}$.

3 Results and discussion

The nucleation treatment and consequent stabilization annealing of low $[O_i]$ samples did not produce any defect luminescence. TEM investigation does not reveal defects at this stage either. However, chemical etching of samples showed a defect density on the level of 10^7 cm^{-3} . A noticeable detection of defects by TEM was observed only after the first steps of high-temperature annealing at 1000 °C. Nevertheless, the nucleation annealing had a strong influence on the intensity of luminescence, developing after subsequent high-temperature annealing. Typical modification of DRL depending on the duration of nucleation at 650 °C for two periods of final growth annealing at 1000 °C is shown in Fig. 1a and b. The dashed lines show the standard position of D1 and D2 lines in plastically deformed Si. It is clearly seen that the obtained spectra differ substantially from the deformation-induced DRL spectrum with concentration of intensity at D1 and D2 bands. The spectral distribution of intensity progressively changes with total annealing time, gradually shifting the band peaks to the D1 and D2 standard positions. However, the effect of high-temperature annealing on the spectral distribution of intensity is much more pronounced than nucleation annealing. It must be noted that considerable differences between spectra are caused by relatively small difference in $[O_i]$.

Figure 2 shows the dependence of DRL spectra on the duration of annealing at 1000 °C.

All the samples had the same nucleation processing at 650 °C. Comparing Figs. 1 and 2 one can see that while nucleation annealing at 650 °C changes the intensity of the spectrum, the prolonged annealing at 1000 °C influences the spectral distribution of intensity as well. While the integral intensity of DRL follows, with rare exceptions the defect density, defined by optical microscopy (Fig. 3), there is no correlation between any particular defect types and DRL intensity.

The defects revealed by TEM can be divided into three main groups: spherical and platelet precipitates without dislocations, platelet precipitates with dislocation loops and isolated dislocation loops, and stacking faults. Typically, our samples contained all three groups of defects. Their relative concentration depended on the duration and temperature of thermal treatment and changed not in a monotonic way. The basic defects at the nucleation stage are small oxygen precipitates with sizes of several tens of nanometers (Fig. 4a). As was mentioned above, no defect luminescence was observed after this stage. Further treatment causes a precipitate growth and sequential dislocation generation. There are two types of dislocation–precipitate conglomerates: linear (LPDC) and globular (GPDC), which are shown in Fig. 4b and c. The later stages of precipitation are attended by formation of stacking faults (Fig. 4d). The absence of a correlation between the luminescence bands and the concentration of particular defects leads to an as-

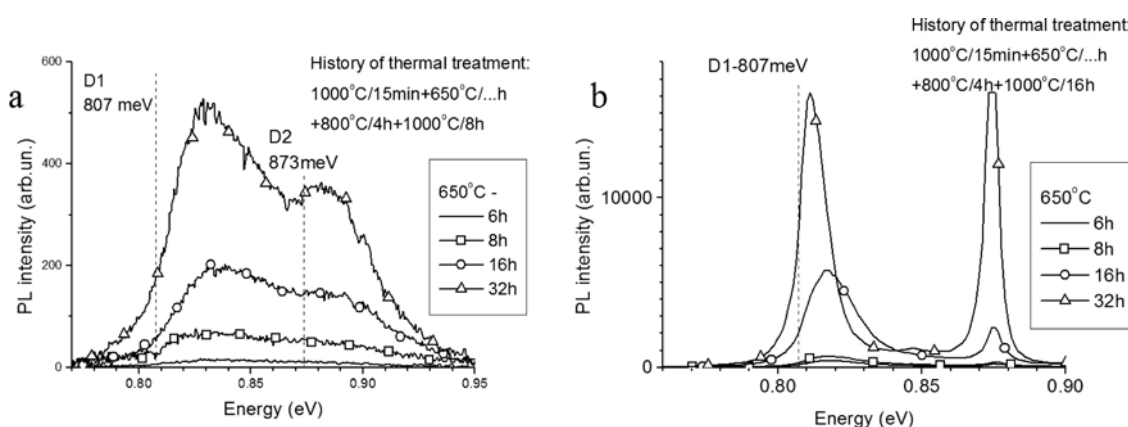


Fig. 1 Two sets of DRL spectra for samples with low $[O_i]$. Each part illustrates the dependence of the spectra on nucleation annealing duration at 650 °C. The (a) and (b) parts correspond to final growth annealing for 8 h and 16 h, respectively.

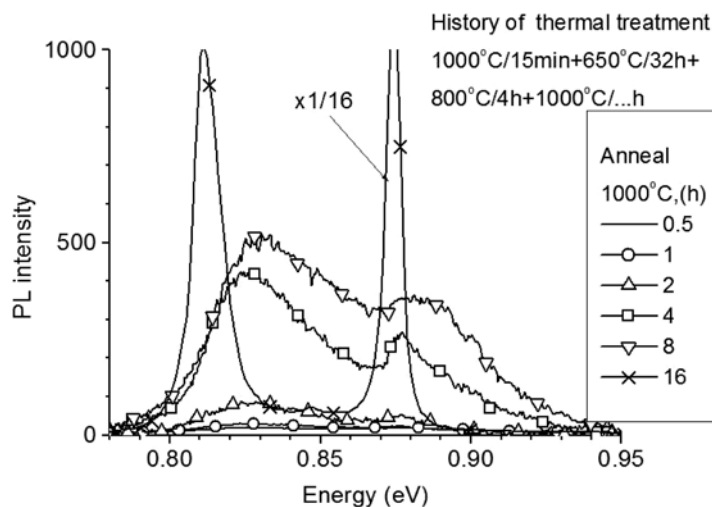


Fig. 2 Dependence of DRL spectra on the duration of growth annealing at 1000 °C.

assumption that the luminescence originates from some secondary defects, which are generated at the primary defects. The gradual transformation of spectra to the well-known DRL spectrum implies that these secondary defects are located on dislocations.

The observed dependence of DRL spectra on thermal processing is similar for samples with different $[O_i]$, but the rate of change of the spectral distribution of intensity was found to be very sensitive to $[O_i]$. Figure 5 shows two examples of DRL spectra and the corresponding TEM images for samples with different initial oxygen concentration. The thermal treatment is identical for samples presented in each part of the figure. The distinction between (a) and (b) is the different duration of growth annealing at 1000 °C, equal, respectively, to 8 h and 16 h. As was mentioned earlier, all types of defects are presented in the samples. Therefore, in Fig. 5 only images of the most typical defects are represented, to compare them. The total concentration of defects for samples with low and medium $[O_i]$ differs by almost two

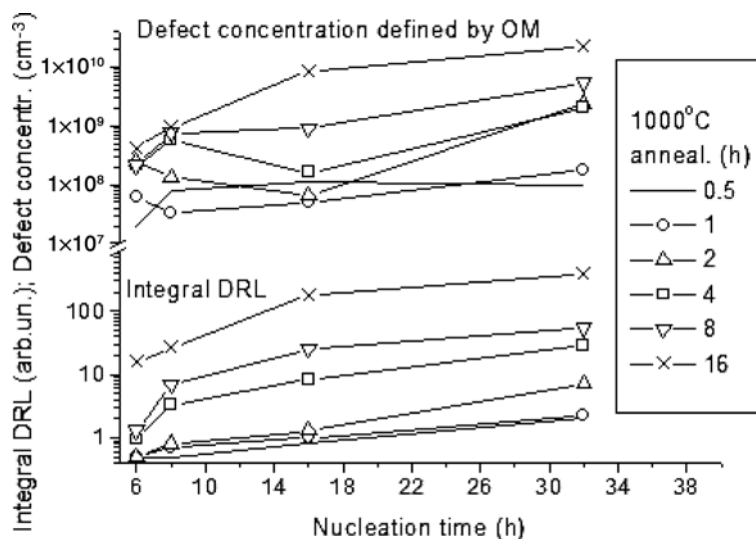


Fig. 3 Comparison of integral DRL intensity and total defect concentration defined by optical microscopy for samples with low $[O_i]$. The x -direction is the duration of nucleation annealing at 650 °C. The curves correspond to different duration of growth annealing at 1000 °C.

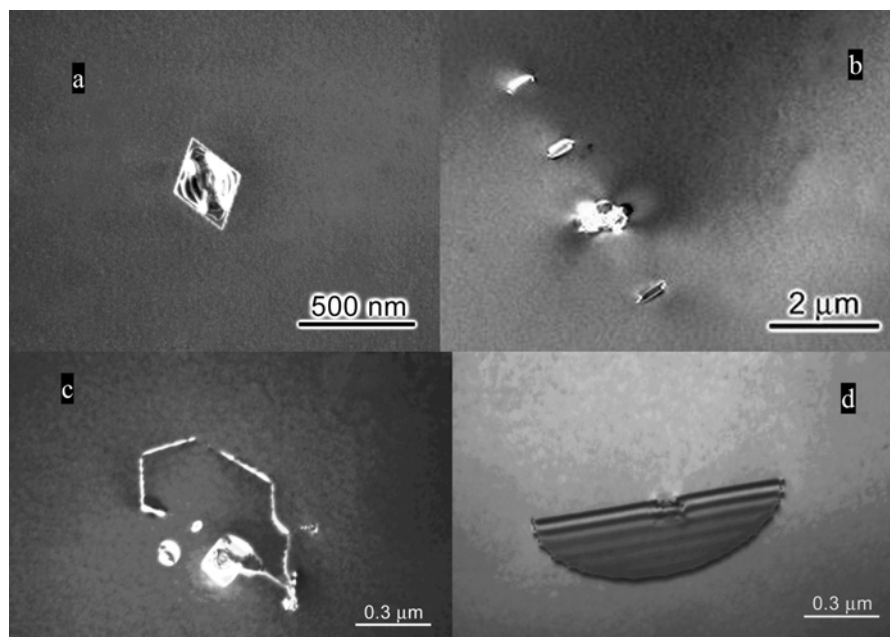


Fig. 4 Typical defects generated in Cz Si due to thermal treatment. (a) – platelet precipitate (PP), (b) – linear precipitate–dislocation conglomerate (LPDC), (c) – globular precipitate–dislocation conglomerate (GPDC), (d) – stacking faults (SF).

orders of magnitude. Actually, we see different stages of the gradual transformation of the DRL spectrum. Again, it started from a broad structureless band. While the left image of Fig. 5a shows a precipitate with punched out dislocation loops, the right image demonstrates oxygen platelet precipitates and perfect dislocation loops not related to the precipitates. The latter result implies that dislocation loops have moved far away from the parent precipitates. Though there is no large difference between the types and sizes of dislocation loops on the left and right images in Fig. 5a, a great difference exists between the related DRL spectra. The intensity of the spectrum of the low $[O_i]$ sample is multiplied by factor of 10 for convenience. The same situation of relative similarity of defects on the left and right images can be seen in Fig. 5b. The difference in intensity between the corresponding DRL spectra in Fig. 5b is less than in Fig. 5a, which is typical for a longer duration of growth annealing at 1000 °C. Nevertheless, the difference in the spectral distribution of intensity is clear. The maximum position of the D1 band in Fig. 5b is notably shifted to the high-energy side for low $[O_i]$ sample.

The fact that dislocation morphology does not differ very much in samples with low and medium $[O_i]$ series, while the DRL spectra differ considerably, leads to a conclusion that the basic transformation of luminescence centers results from microscopic interaction between structural defects on dislocations and oxygen atoms or clusters. Looking at the spectra of Fig. 2, we see that 16 h of growth annealing is not sufficient to get the standard position of the D1 line at 807 meV. However, additional annealing for only 3 h is enough to shift the D1 line maximum close to a final position of 807 meV (Fig. 6).

On the other hand, low-temperature annealing of the same sample leads to the shift of the D1 band in the opposite blue direction (Fig. 6). The latter behavior can be compared with the spectral distribution of intensity change in plastically deformed samples after quench and subsequent low-temperature annealing (Fig. 7). This type of annealing is known to collect oxygen around dislocations in the form of individual atoms or small clusters [13] but it is not enough for forming large precipitates [14].

The luminescence in samples with high $[O_i]$ demonstrates a somewhat different behavior. Even after short annealing at high temperature the DRL spectra resemble the spectra of medium $[O_i]$ samples after several hours annealing at 1000 °C. Apparently, the rate of precipitate growth is much higher in this

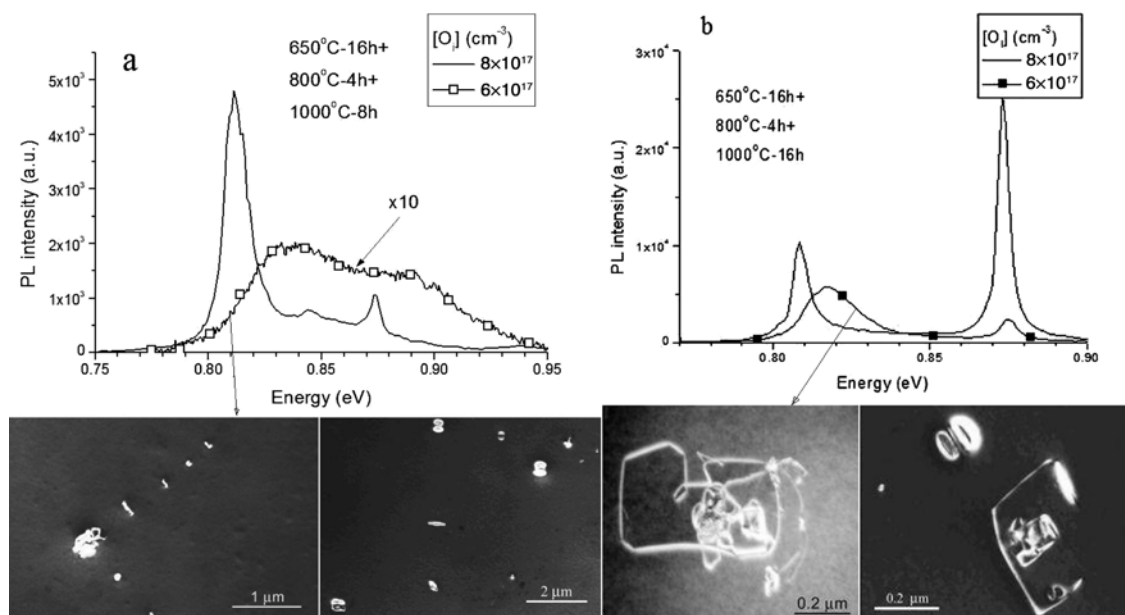


Fig. 5 (online colour at: www.pss-a.com) DRL spectra and TEM images for samples with different initial oxygen concentration. The preliminary nucleation treatment of samples is identical, the only distinction between (a) and (b) is different duration of growth annealing at 1000 °C (shown in the figure). Left and right images related to the samples with low and middle $[O_i]$. Arrows show the correspondence between graphs and images. The detailed explanation is in the text.

case. Indeed, it is reasonable to expect much higher local concentrations of oxygen near dislocations and more intensive decay of the oxygen solid solution in such samples. Figure 8 illustrates the modification of DRL spectra for the sample with high $[O_i]$ after successive anneals at 950 °C. Again, we observe a gradual narrowing and shift of D1 band to the 807 meV position. Unlike low and medium $[O_i]$ samples, the initial broad bands did not appear here.

In addition to “conventional” nucleation treatment the nucleation by the creation of elevated vacancy concentration has been carried out. TEM investigation revealed no substantial difference of defect morphology between conventional and MDZ samples. However, it should be noted that the time necessary

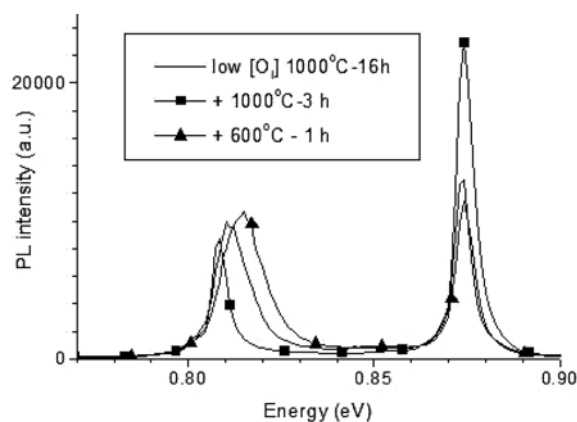


Fig. 6 Opposite shift of the D1 maximum after complementary annealing: squares – at 1000 °C for 3 h, up triangles – at 600 °C for 1 h.

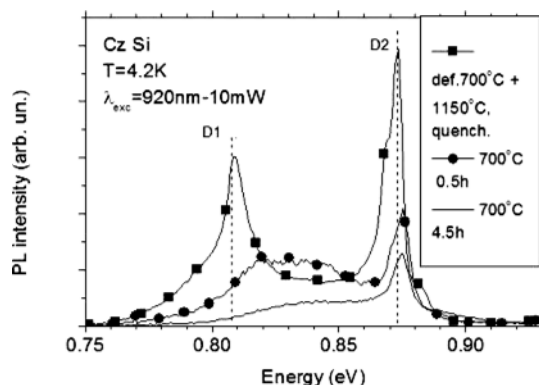


Fig. 7 Blueshift and broadening of the D1 band due to annealing at 700 °C.

for the creation of a certain defect density in MDZ samples is several times shorter than a corresponding growth periods for conventional samples. Figure 9 shows a modification of the DRL spectra for medium $[O_i]$ samples on the growth duration at 1000 °C. In contrast to Fig. 2, where the noticeable shift of the D1 band happens only at the last annealing step, the gradual shift of D1 maximum started in the MDZ samples from the very beginning of annealing.

Following our previous model, we must assume that the punching-out process starts in MDZ samples at the initial stages of high-temperature annealing. TEM investigation supports this assumption. In conventional samples, the growth of precipitates started from small spherical precipitates and later platelet precipitates of small sizes (30–50 nm). As growth annealing continues, spherical precipitates disappear and the sizes of platelet precipitates gradually increase up to 80–120 nm. After that, the process of dislocation punching out starts with creation of LPDC. In MDZ samples, no small precipitates have been observed even after short growth annealing due to the effective decay of solid solution. The basic defects in such samples are platelet precipitates of sizes 100–120 nm, which form LPDC. Thus, dislocations in MDZ samples are exposed to high temperature annealing from the very beginning of the growth stage, causing a gradual transformation of DRL spectra.

Considering all these observations we can suggest the following scenario for formation of luminescence centers responsible for DRL during the multistep anneals of Cz Si. The source of luminescence is

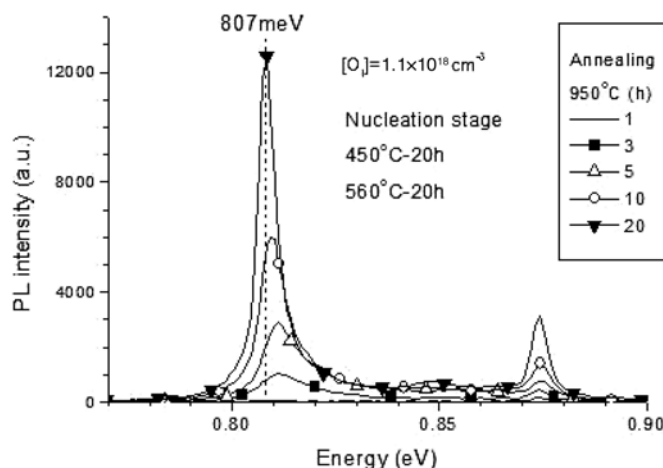


Fig. 8 Modification of DRL spectra due to gradual growth of precipitates after successive annealing at 950 °C.

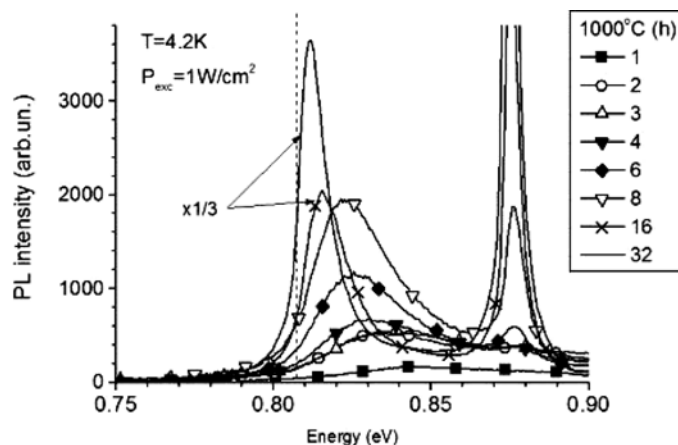


Fig. 9 Modification of DRL spectra after successive annealing at 1000 °C. Curves 16 h and 32 h were diminished by 1/3 to show details of the initial spectra.

defects on dislocation loops developed around precipitates. In the initial stages of formation of dislocation loops, large numbers of oxygen atoms and small clusters surround dislocations. The DRL spectra look at this stage as broad unresolved bands the intensity of which is proportional to the number of precipitate–dislocation conglomerates, which in turn depends on the duration of nucleation treatment. An expansion of dislocation loops is limited by stress fields around precipitates. Therefore, at some moment they stop at a final position. After this, the continuing high-temperature annealing stimulates the process of redistribution of oxygen along dislocation line, which dominates other diffusion-limited processes [13]. Namely, oxygen atoms and small clusters uniformly distributed along dislocation lines conglomerate gradually to larger and larger precipitates. In other words, a redistribution of oxygen near to and at dislocation lines leads to transform from a large number of small precipitates and clusters to a lower number of large precipitates and can be considered as a “cleaning” of some parts of dislocations from the oxygen atmosphere. A direct spectroscopic effect of this process is a gradual movement of the maximum position of the D1 band to the standard position of 807 meV. It must be noted that the “cleaning” process can also be produced by repulsion of dislocations from large precipitates, when the sizes of secondary precipitates exceed some critical dimension [14]. In this case, dislocations break away from surrounded oxygen atmosphere. However, we have never observed such a phenomenon in our samples, while a prime growth of secondary precipitates on dislocations has been often detected, especially for samples with medium and high $[O_i]$. The annealing-dependent shift of the D1 band has been reported earlier for precipitated samples [15] and plastically deformed samples [5]. From this point of view, the result of Fig. 5 looks quite natural. In the samples with medium $[O_i]$ the formation of dislocation loops has been completed much earlier than in samples with low $[O_i]$. Therefore, for an equal duration of heat treatment of two types of samples, in medium $[O_i]$ samples dislocations were annealed for a longer period and redistribution of oxygen proceeded much further there. The same process occurs in samples with high $[O_i]$, but it goes much faster. Taking into account that the precipitation process begins with formation of small clusters, and the rate of their formation has a power dependence on the initial oxygen concentration as a function of anneal temperature [16], we can easily suggest that the precipitation process around dislocations is strongly accelerated with increase of $[O_i]$. What is more, the local concentration of oxygen around dislocations is supposed to be much higher than the bulk concentration due to the high gettering efficiency of dislocations [17].

The open question still remains, what is the nature of the initial broad bands? In Refs. [10, 11] a model of carrier confinement in a strain-induced field has been suggested. The theoretical evaluation of band-gap shrinkage around disk-shaped precipitates is not at variance with the model [11]. According to the above results it is apparent that oxygen plays a primary role in transformation of broad bands. In our

opinion, gradual conversion of a long-wave broad band to the D1 line witnesses participation of the same dislocation-related center in transitions. In that case an interaction with oxygen causes a broadening and a shift of transition energy. We can assume two possible ways of interaction of oxygen with DRL centers: elastic and coulomb interaction. The former assumption looks unlikely, because elastic interaction should influence on all dislocation-related centers. At least we can expect the same behavior of the D2 center, the properties of which are similar to D1 properties in many respects. What is important is that the response of these two lines to a uniaxial stress is identical [7]. But our results clearly show that this is not the case (e.g. Figs. 8 and 9). In previous papers we suggested a recombination between thermal donors (TD) and dislocation acceptors to explain the appearance of broad shoulders near the D1 line [5, 8]. In the case of FZ Si this model allowed the position and shape of the longwave shoulder of the D1 line to be described consistently [8]. In Cz Si the shoulder has a blueshift relative to D1 line. This can be explained by participation of different type of TDs with ionization energy less than usual TDs. Indeed, the generation of so-called new TDs has been observed after annealing of Cz Si at temperatures above 600 °C [18]. Therefore, there is a good reason to believe that the broad bands, arising after the first steps of high-temperature annealing originate from DRL centers perturbed by interaction with oxygen atmosphere.

In conclusion, it must be noted that the effectiveness of dislocations in the sense of DRL intensity in precipitated samples is sufficiently higher than those in plastically deformed samples. The rough estimation of the common length of dislocations at precipitate densities of 10^9 cm^{-3} gives a value of about 10^6 cm , which is at least two orders less than the corresponding dislocation density in plastically deformed samples with the same intensity of the D1 line. This implies that either the line density of D1 centers on dislocations is higher in precipitated samples or recombination conditions near precipitates favor the effectiveness of radiative recombination in this case. Anyway, the generation of dislocations by the precipitate growth can be considered as an alternative and competitive way for preparation of emitting structures. The preliminary study of temperature quenching of the D1 band in precipitated samples as compared to plastically deformed samples showed that up to temperatures of about 130 K the relative intensity of the D1 band remains higher in precipitated samples (Fig. 10). The following crossover of curves can be caused by the following reason: the spatial distribution of dislocations in plastically deformed samples is more or less uniform after high-temperature deformation ($>900 \text{ °C}$), while the distribution of dislocations in precipitated samples is modulated by the precipitate sites. The local dislocation density around precipitates can reach values of about 10^{12} cm^{-2} and is very low in-between precipitates. That is an advantage of a precipitation-generated dislocation structure due to the nonlinear dependence of the relative intensity of the D1 band on dislocation density [6]. But it also can be a reason for high-

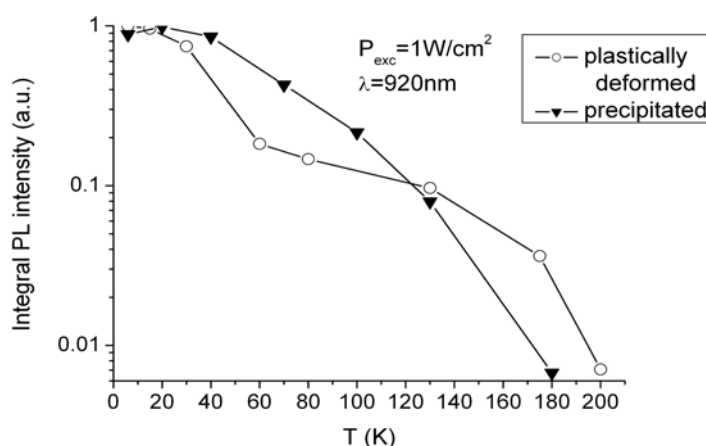


Fig. 10 Temperature dependence of the integral intensity of the D1 band in plastically deformed and precipitated samples. For convenience, curves are normalized to maximum intensity at low temperature.

temperature quenching of dislocation radiation in precipitated samples due to participation of excitons in capture processes other than the trapping by dislocations. The preparation of a more uniform distribution of dislocations in precipitated samples is a further task of this activity.

4 Conclusions

Summing up the above results, we can say that multistep annealing of Cz Si samples provides an effective generation of dislocations. The intensity of DRL depends on the nucleation period, when a site density of future precipitates is created. The spectral distribution of intensity of DRL depends mainly on the final high-temperature annealing. As a result, the whole intensity of the D1 band can be concentrated in the narrow line with position at 807 meV and the relatively high intensity of the D1 line can be achieved by this technique. This transformation happens mainly due to much higher local dislocation density reached in precipitated samples. The preliminary RTA treatment of the samples allows reduction of the whole period of thermal treatment without a loss of DRL intensity.

The intensity of the more promising D1 band is much higher at low temperatures in precipitated samples with equal mean dislocation density than in plastically deformed samples. Therefore, it confirms the competitiveness of the generation of dislocations by the precipitation process for LED application.

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