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The photoluminescence emission in the 0.7–0.9 eV range from oxygen precipitates, thermal donors and dislocations in silicon

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Abstract. There is a wide set of literature reports that suggest that over-coordinated oxygen or self-interstitials are, directly or indirectly, the chemical bridge between thermal donors, oxygen precipitates and dislocations, capable of supporting a common origin of their emission features in the 0.7–0.9 eV range. Finding the experimental proof of these suggestions was the aim of this present work, which required both appropriate preparation of samples and their careful optical, electrical and microscopical characterization.

We were able to show not only that the photoluminescence emissions from oxide precipitates could be correlated to their density and to the presence of closed dislocation rings around them, but also that the precursors of dislocations are optically active as well. For samples thermally annealed in the range of thermal donors, we were able to show that their optical activity seems to be correlated to a transition from a shallow donor level of thermal donors to a deep level of a C_iO_2 complex.

1. Introduction

It is well known [1–10] that a number of photoluminescence bands due to (or associated with) the presence of dislocations in silicon (four main bands, conventionally labelled D1–D4, where $E_1 = 0.807$ eV, $E_2 = 0.870$ eV, $E_3 = 0.95$ eV and $E_4 = 0.99$ eV), to C_i–O_i complexes (line C at 0.789 eV) [11] and to C_i–O_2 complexes [12] or, possibly, to thermal donors (P line at 0.767 eV) can be observed in the energy range 0.7–0.9 eV at 12 K. In addition, photoluminescence from oxygen precipitates [13] has been shown to originate in the same region as the dislocation luminescence.

At a first glance, it seems difficult to find some common structural, physical or chemical properties of these defects capable to explain their emission features in this energy range. However, it is worth remarking that at temperatures as low as 450 °C (temperature range of thermal donors) dislocations generated by plastic deformation at 650–700 °C do interact with oxygen [9]. The result of this interaction is the quenching of the D2 luminescence in favour of that of oxygen-related centres emitting at 0.778 eV and at 0.850 eV, showing a possible inter-relation between dislocations and thermal donors (TDs).

Apparently, dislocations passivated with oxygen do behave, in the temperature range of TDs, as heterogeneous catalysts for the conversion of TDs (whose presence is always associated with an emission at 0.767 eV) to oxygen clusters which luminesce in a very close energy range, while simultaneously quenching the dislocation luminescence.

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On the other hand, it is well known that the growth of silicon oxide from silicon oxide embryos at temperatures as high as 800 °C, or more, occurs with the simultaneous emission of self-interstitials, which, after clustering, are the precursors of dislocations.

The hypothesis that aggregates of self-interstitials injected into the solid matrix by growing oxygen precipitates are at the origin of the D1 and D2 luminescence was proposed recently by one of the present authors [13], on the basis of the experimental dependence of the energy of the D1 and D2 bands on the size of oxide precipitates nucleated and grown in carefully prepared samples. A similar hypothesis has been also suggested by Jones *et al* [14] on the basis of theoretical considerations about the optical properties of the silicon hexavacancy and of multi-interstitial defects, which according to the authors may arise from oxygen precipitation.

It is therefore possible to suggest a common link between oxygen precipitates, dislocations and thermal donors, although certain experimental proof of a correlation between the photoluminescence emission at 0.767 eV (P line) and the thermal donor concentration is still lacking, leaving the doubt that the P line is not due to TDs. In fact, only the annealing time dependence of the intensity of TD-bound exciton emission in the TO range was sought and demonstrated to exist [15], as the TDs were not considered the possible source of the emission at 0.767 eV.

The physical background of this conclusion is that the P line, when detected in carbon-rich silicon wafers irradiated with electrons and thermally treated at 470 °C, shows ^{13}C and ^{14}C isotope shift [12, 15] and is due, therefore, to a C-O complex having the configuration of a C_i–O_2 species [12]. In turn, the formation of a C_i–O_2 complex arises from the reaction of a di-oxygen complex with a interstitial carbon [12].

A challenging suggestion about the existence of a property correlation among thermal donors and oxide precipitates comes however from the conclusions of the theoretical modelling of atomic dynamics of the nucleation, growth and annealing of thermal donors and of the growth of silicon oxide in a silicon matrix [16]. This model is consistent in both cases with the presence of over-coordinated oxygen in a metastable configuration, which reaches a stable configuration with the emission of a silicon self-interstitial.

It turns out that over-coordinated oxygen and/or self-interstitial clusters could be responsible for some of the PL emission of thermal donors and of oxygen precipitates. This last hypothesis is indirectly substantiated by *ab initio* calculation of the structure of C_i–O_i complexes [17], characterized by the emission at 0.789 eV (C line), very close to that of the P and D1 and D2 bands, whose vibrational properties can be modelled by assuming the presence of over-coordinated (frustrated) oxygen atoms.

Some other information on defect centres in thermally treated silicon or dislocated silicon comes from DLTS spectroscopy and Hall effect measurements.

From DLTS experiment, Kimerling and Benton [18, 19] found in fact in CZ silicon thermally treated at 470 °C two donor defect states at $E_c - 0.07$ eV (TD^0) and $E_c - 0.15$ eV (TD$^+$), which are ground states of helium-like, effective mass centres, presenting the properties of a local dipole from field assisted ionization experiments.

Also from DLTS measurements, four centres labelled A, B, C and D are identified in nominally clean float zone silicon dislocated at 650 °C [20–22] of which that at $E_c - 0.4$ eV (C centre) is shown to be associated with the dislocation core [22]. Finally, Hall effect measurements on CZ silicon annealed at 470 °C for two hours did allow identification of a deep centre at $E_v + 0.35$ eV [23].

There is therefore a wide set of indications that suggest that over-coordinated oxygen or self-interstitials are, directly or indirectly, the chemical bridge between thermal donors, carbon–oxygen complexes, oxygen precipitates and dislocations, capable of supporting a common origin of their emission features in the 0.7–0.9 eV range. Finding an experimental proof of this suggestion was the aim of this present work.

2. Experimental details

Two types of sample were used for these experiments.

The first set, used for investigating the luminescence of oxygen precipitates, consists of oxygen segregated silicon samples extracted from a matrix already systematically studied by Borghesi *et al* [24], prepared in VLSI conditions by MEMC Electronic Materials. They were systematically submitted to sequential annealing processes (grown-in precipitate dissolution for 15 min at 1000 °C to achieve constant starting conditions + nucleation at 650 °C + precipitation at 800 °C + 1000 °C) with variable nucleation (4, 8, 64, 120 hours) and precipitation (4 hours at 800 °C and 4, 8, 32, 64 hours at 1000 °C) times.

The samples were characterized for oxygen losses and microprecipitates (bulk micro-defects, BMD) density using both FTIR measurements and etching techniques followed by optical microscopy measurements, while transmission electron microscopy (TEM) was used for microstructural characterization, including dislocation identification.

The second set, used for the study of the luminescence features of thermal donors, consists of samples of CZ p- and n-type silicon which were treated for variable times from 8 to 98 hours at 470 °C in order to induce the thermal donor generation. They were cut from 6 in diameter wafers (MEMC Electronic Materials, Italy) with an initial oxygen content, measured by conventional FTIR measurements, around 7×10^{17} at.cm^{-3}. Their carbon content was found to be below the FTIR detection limits ($<10^{16}$ at.cm^{-3}).

Prior to thermal treatments, the samples were RCA etched to polish the back side and to remove unintentionally deposited metallic impurities on the front side, and then rinsed with ultrapure Merck distilled water. After the etching and cleaning procedure, the samples were closed under vacuum in quartz ampoules, each of which contained one p and one n sample. Thermal treatments were carried out in a horizontal furnace at 470 °C. The extraction of the ampoules was made manually and the quenching time was less than 30 s.

The photoluminescence spectra of both sets of samples were recorded with a spectral resolution of 3 nm, using standard lock-in techniques in conjunction with a grating monochromator and InGaAs as a detector. For the excitation, a quantum well laser ($\lambda = 808$ nm) with a power density of 6 W cm^{-2} was used. The measurements were performed in a variable-temperature cryostat in the temperature range 12–300 K. The effect of the temperature and of laser power on the position and intensities of the PL bands was also investigated in specific cases.

The concentration of thermal donors in the second set of samples was determined from the results of resistivity measurements at room temperature, carried out using the four point method.

3. Experimental results

3.1. Optical and microstructural properties of oxygen precipitates

In a previous paper [13] we have already presented some preliminary results of an extended study dealing with the PL of oxygen precipitates, obtained with the shortest (4 hours) nucleation annealing stage at 650 °C. We could demonstrate (see figure 1) that in these samples the onset of a strong D1 and D2 luminescence occurs after annealing at 1000 °C, even before any significant decrease of the interstitial oxygen. The intensity of both lines was shown to increase with the precipitation time at 1000 °C, and then with the increase of the size of precipitates. The D3 and D4 luminescence was systematically below the detection limit.

These present results show that the D1 and D2 lines are still the most prominent spectral features of all the samples investigated. In fact, the D3 and D4 bands are undetectable even at

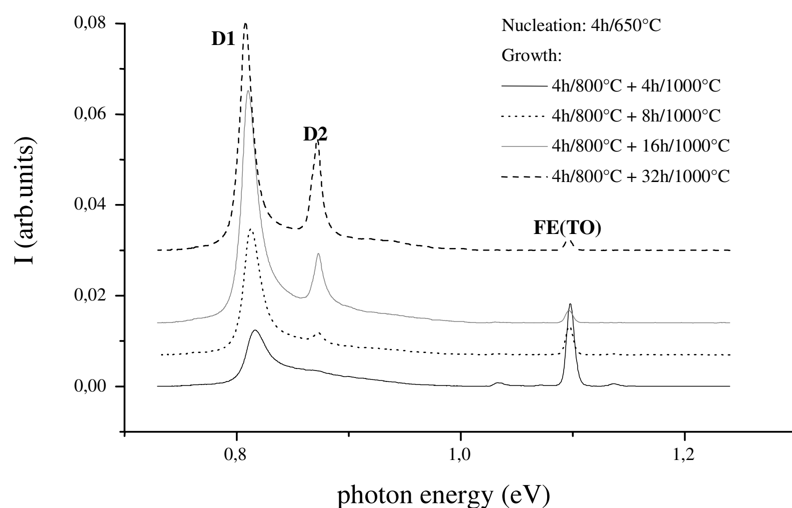


Figure 1. Photoluminescence spectra of CZ silicon subjected to short nucleation anneals at 650 °C.

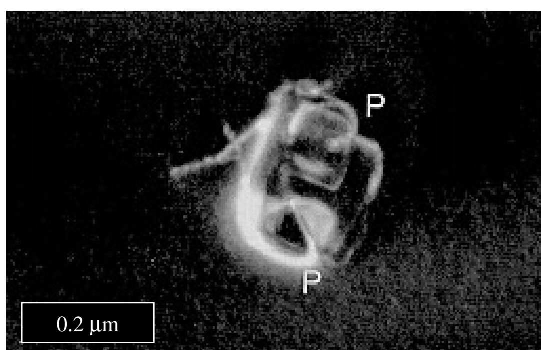


Figure 2. TEM micrograph of a couple of precipitates associated to dislocations: the square form of the precipitates suggests that they are octahedral in shape.

the longest precipitation anneals, or in samples submitted to long nucleation anneals, unless the precipitation stage at 1000 °C is longer than 64 hours for 8 hours of nucleation, 32 hours for 64 hours of nucleation and 16 hours for 120 hours of nucleation.

From TEM investigations carried out on a sample subjected to a long nucleation (64 h) and precipitation annealing (4 h at 800 °C and 64 h at 1000 °C) it was possible to show (see figures 2 and 3) that precipitates are always associated with dislocations and that dislocations are often decorated with small precipitates.

When the D3 and D4 bands are present (see figure 4, where the PL spectrum of the sample subjected to TEM investigation is displayed) the overall spectral features look qualitatively very similar to those observed in plastically deformed samples [7]. As shown in figures 5(a) and (b), a significant dependence (more than one order of magnitude) of the intensity of the D1 and D2 lines is observed as a function of the nucleation and precipitation time, with a maximum always lying at 8 hours of nucleation and 16 and 32 hours of precipitation annealing at 1000 °C, for the D1 and D2 line, respectively. It is also quite remarkable that in these samples, presenting a total density of about 10^{11} microdefects cm^{-3} (less than 10^7 cm^{-2}), the

Figure 3. TEM micrograph of precipitates (P) and planar defects (D), consisting of dislocations decorated by small precipitates.

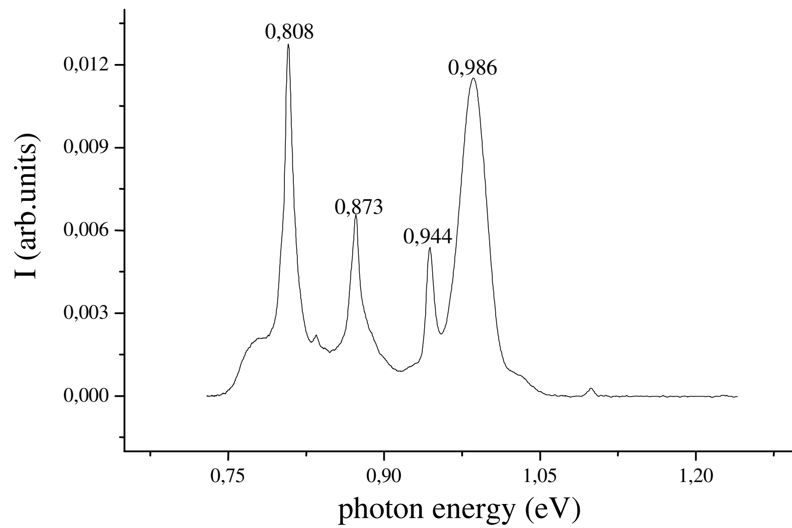


Figure 4. Effect of long nucleation and precipitation anneals (64 h at 650 °C + 4 h at 800 °C + 64 h at 1000 °C) on the photoluminescence spectra of oxygen precipitates.

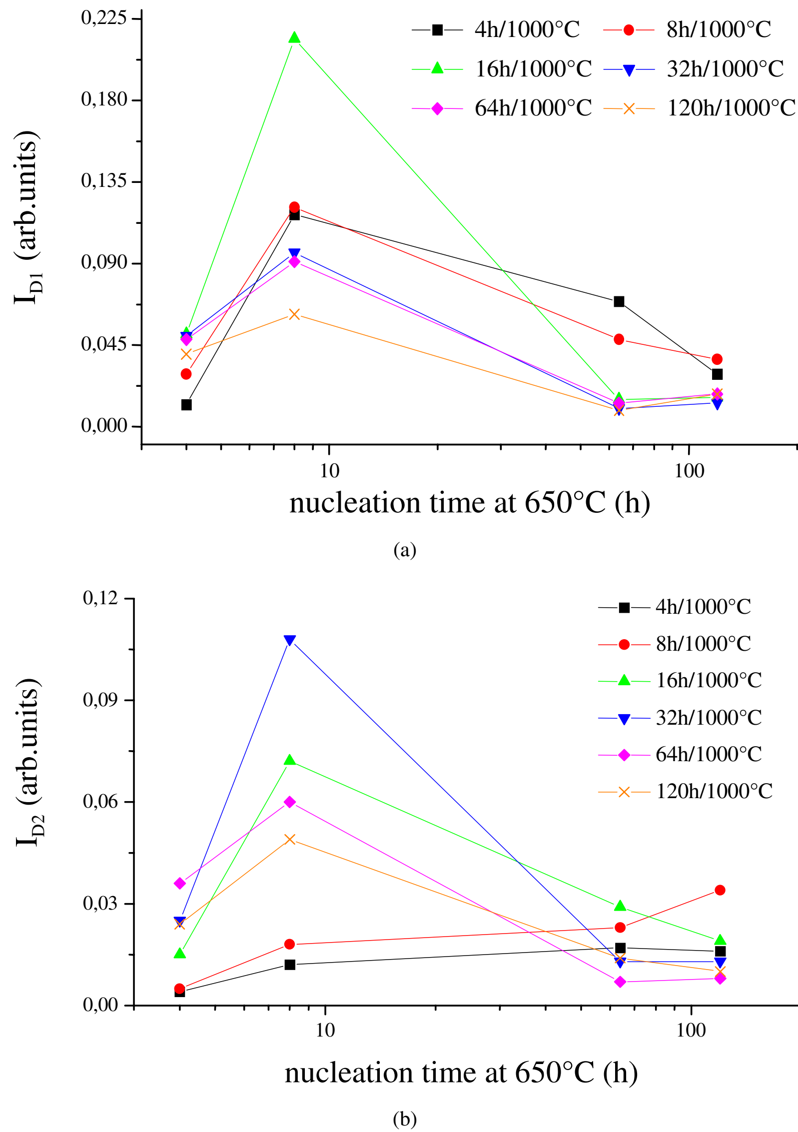


Figure 5. (a) Dependence of the intensity of the D1 line on the nucleation and precipitation time. (b) Dependence of the intensity of the D2 line on the nucleation and precipitation time.

intensities of the D1 and D2 bands are at least one order of magnitude larger than those of plastically deformed silicon with a dislocation density larger than 10^7–10^8 cm^{-2} [7].

The difference, as found by TEM investigation, is not only that precipitates are associated with dislocations, but also that dislocations take the form of closed (figure 2) or hexagonal (figure 6) rings around the precipitates.

Another notable difference appears in samples subjected to short nucleation (up to 16 h) and precipitation anneals. The position of the D1 line (literature value, 0.807 eV in dislocated samples), in fact, is shifted towards higher energies (up to a maximum of 0.818 eV) and shows a monotonic decrease towards an asymptotic value of 0.807 eV with the increase of both the nucleation and precipitation time (see figure 7 and 8).

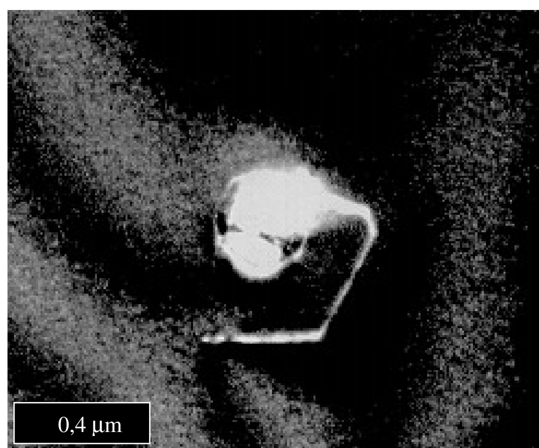


Figure 6. TEM micrograph of an oxide precipitate associated with a dislocation.

A similar behaviour is shown by the D2 band (figure 9), where the trend, however, seems to indicate a tendency to an energy decrease below the literature value.

The duration of the nucleation and precipitation anneal is very influential on the volume of the precipitates (given as the number of oxygen atoms/precipitate), as shown in figure 10, for a short and a long nucleation time. It appears that for the sample submitted to a long (64 h) nucleation anneal the growth kinetics follow monotonically Ham's law, which predicts a linear dependence on the square of the radius of the precipitates:

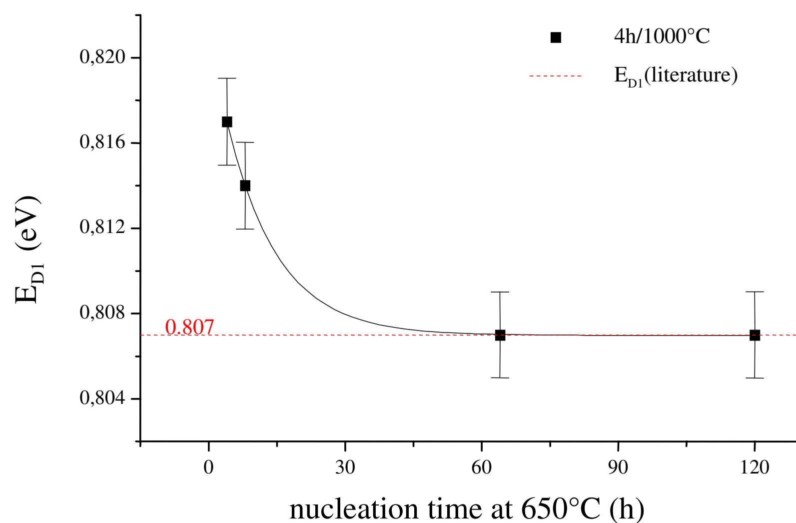
$$r^2(t) \cong 2D[(c_0 - c')/(c_p - c')]t$$

(where c_0 is the initial oxygen concentration, c_p is the oxygen concentration in the precipitate and c' is the concentration of oxygen at the precipitate/matrix interface) and, then, a volume increase with $t^{3/2}$ [25]. Instead, for the sample submitted to a short (4 h) nucleation anneal an initial much faster growth rate is observed. The different slope of the two curves might be accomplished by the different initial oxygen concentration as well as by a possible difference in the stoichiometry of the oxide precipitates.

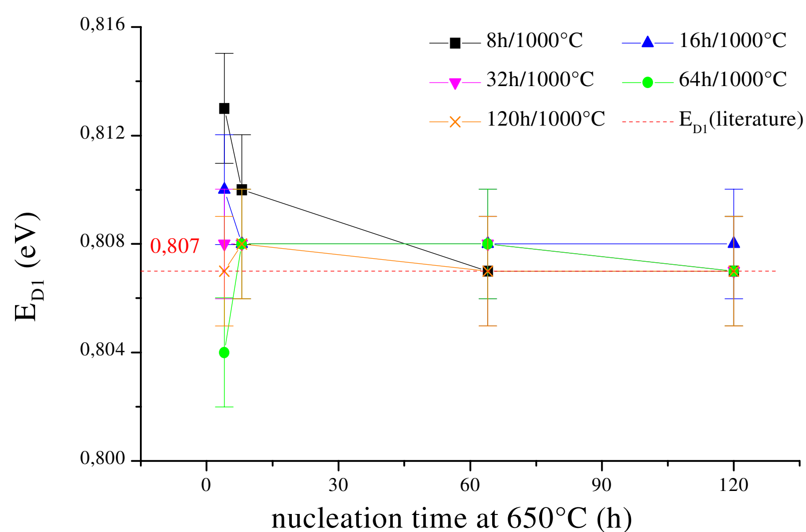
3.2. Photoluminescence of silicon in the range of thermal donors

Figure 11 reports a typical luminescence spectrum of a sample annealed at 470 °C, which shows a very intense P line at 0.767 eV, and two weak signals attributed to the C (0.789 eV) and H (0.925 eV) bands, in addition to the bands of TD-bound excitons [15], which were deliberately not studied in this work.

While the energy position of the P line is demonstrated to remain unaltered with increasing annealing times and by increasing the laser power within four orders of magnitude (from 0.02 mW to 60 mW), its intensity shows a typical trend (see figure 12) for both the p-type and n-type samples, with the duration of the heat treatment. The intensity of the P line, increases, in fact, up to a maximum after about 25 h, and then decreases for thermal treatments of longer duration, the decrease of B-doped samples being faster than that relative to P-doped samples. It is worth remarking that this behaviour is largely uncorrelated with the total concentration of TDs, which increases steadily, but follows almost exactly the behaviour of the TD-bound exciton emission in the TO range, which peaks in fact at about 20 hours [15].



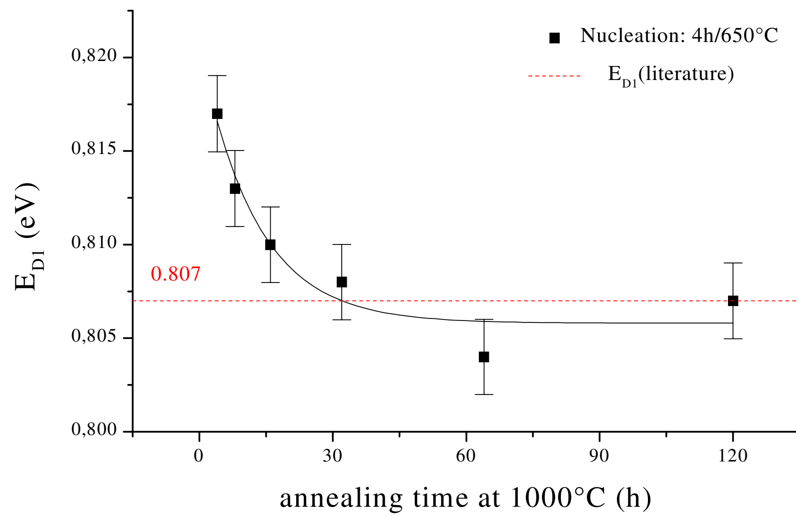
(a)



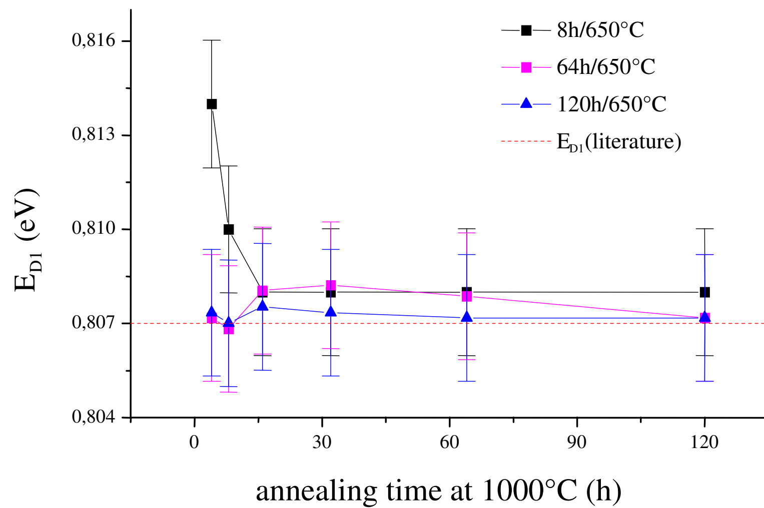
(b)

Figure 7. (a) Dependence of the energy position of the D1 line as a function of the nucleation time at 650 °C for a sample subjected to a short (4 h) precipitation anneal at 1000 °C. (b) Dependence of the energy position of the D1 line as a function of the nucleation time at 650 °C for different precipitation anneals at 1000 °C.

Eventually, for heat treatments of 98 hours or more, in agreement with the literature [11, 26], the photoluminescence spectrum consists of a broad band between 0.9 eV and 1.1 eV and of two lines belonging to the O_i series (O_1 at 1.118 eV and O_2 at 1.116 eV). In all circumstances, the intensities of the C and H lines remain uninfluenced by the duration of the heat treatment.



(a)



(b)

Figure 8. (a) Energy position of the D1 line as a function of the precipitation time at 1000 °C for a sample subjected to a short (4 h) nucleation anneal at 650 °C. (b) Energy position of the D1 line as a function of the precipitation time at 1000 °C for different nucleation anneals at 650 °C.

4. Discussion

4.1. Photoluminescence of oxygen-precipitated silicon

The high intensity of the PL signals of the oxygen precipitated samples could be at least preliminarily explained by supposing that it is due to dislocations associated with precipitates. In the presence of a closed dislocation ring which includes pure edge, pure screw and mixed dislocation segments, each precipitate might in fact behave as a localized source of luminescence.

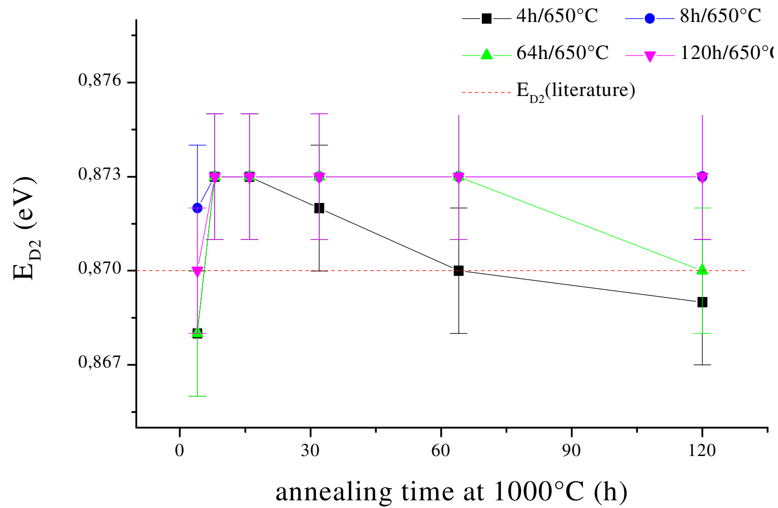


Figure 9. Energy position of the D2 line as a function of the precipitation time at 1000 °C for different nucleation anneals at 650 °C.

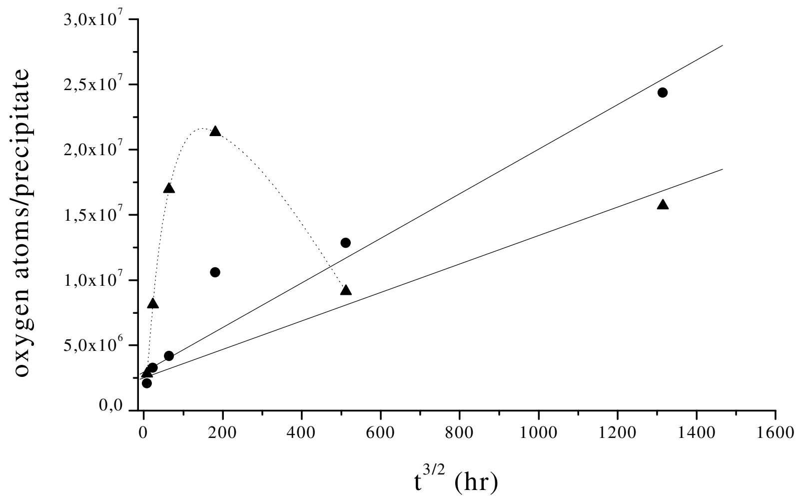


Figure 10. Oxide precipitate volume evolution during thermal annealing at 1000 °C (▲ nucleation at 650 °C for 4 hours, ● nucleation at 650 °C for 64 hours).

Based on the results of the work of Cavalcoli *et al* [22], who showed that among the four deformation-induced traps found by DLTS experiments on dislocated silicon, only the trap C (which lies at $E_c - 0.40$ eV) is the most spatially localized at dislocations, the previous conclusion does not fit entirely with our results: first, because a transition from the C centre to the valence band will account neither with the position of the D1 nor of the D2 line; further, because the energy shift of the D1 and D2 bands observed at the shortest nucleation times (see figure 7 and 8) directly brings into consideration that the precipitates themselves (or the interface between the oxide and the silicon matrix), or deformation-induced point defects around them, more than the dislocations, are at the origin of the oxygen precipitate's luminescence. The largest energy shifts for the D1 band are in fact observed in close

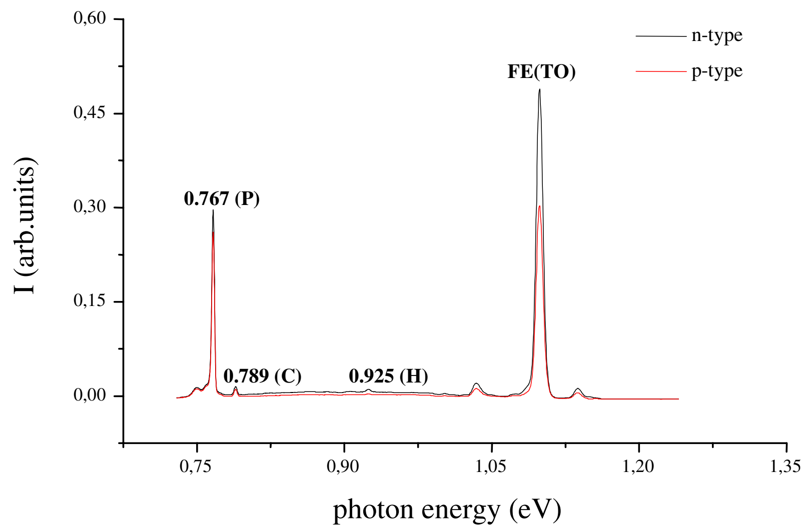


Figure 11. Typical photoluminescence spectra of a p-type and n-type CZ sample annealed at 470 °C.

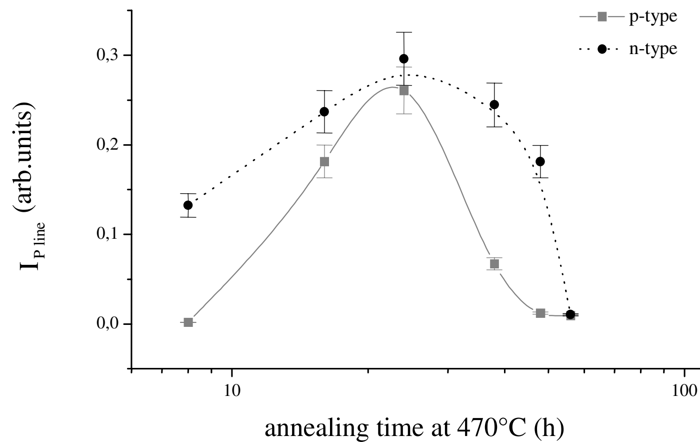


Figure 12. Dependence of the intensity of the P-line luminescence on the annealing time at 470 °C.

correspondence to the formation of anomalously large precipitates, growing with a kinetics which does not follow the Ham law, as was observed in figure 10.

This hypothesis is supported also by the different dependence of the intensity of the D1 and D2 luminescence on the nucleation and precipitation time, and then on the precipitate size, shape and density, as shown in figures 5(a) and (b). In fact, the shift of the luminescence intensity maximum towards larger precipitation times for D2, whose intensity is always lower than that of D1, suggests that the D1 and D2 bands would correspond to the emission of different types of precipitate [25]. Furthermore, it appears also that the absolute intensity of D1 line increases by about a factor of ten by doubling the nucleation time, which in fact results in an increase of a factor of ten (from 1.8×10^{10} to 1.9×10^{11} cm^{-3}) in the BMD for samples nucleated for 4 and 8 hours at 650 °C and annealed for 16 hours at 1000 °C (see figure 5(a)).

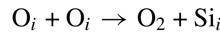
We suggest therefore that the D1 and D2 luminescence observed in oxygen-precipitated silicon is associated with the very properties of the oxide precipitate interface, and, therefore, either with the presence of frustrated oxygen centres, stacking faults or self-interstitial clusters, generated by the interstitials injected into the silicon matrix during the oxide particles' growth.

4.2. Photoluminescence of silicon samples annealed in the temperature range of thermal donors

As a significant carbon isotopic shift (0.079 MeV) is observed on the transition at 0.767 eV (P line) [12], it would be straightforward to conclude that the annealing time behaviour of the P line is associated with the evolution of the C_i–O_2 complex concentration with the annealing time at 470 °C.

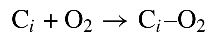
This conclusion, however, does not fit with the results of [15] about the annealing time evolution of the TD-bound exciton luminescence, unless we assume that a thermodynamic equilibrium exists between the C_i–O_2 complexes and the TDs, both generated by annealing at 470 °C.

This last condition would be accomplished if the reactions of TD and C_i–O_2 formation are intimately coupled by a self-interstitial generation and trapping process, where TDs behave as self-interstitial sources [16] and carbon as a self-interstitial sink. This would not be the case when, according to the literature [12], the C_i–O_2 formation process is described by the sequence of reaction



$$O_i + O_i \rightarrow O_2 + Si_i$$

$$Si_i + C_{si} \rightarrow C_i$$



$$C_i + O_2 \rightarrow C_i\text{–}O_2$$

as here there is no possibility of coupling the TD and C_i–O_2 formation processes.

A possible, alternative, solution of this problem arises from our results on the insensitivity of the energy position of the P line to the light intensity, which implies that the transition could not be of the donor–acceptor type, as in this case we should observe a shift of the energy of transition with the light intensity [27].

Instead, a transition from the donor level at $E_c - 0.07$ eV of TD [18] to the deep centre at $E_v + 0.35$ eV observed by Hall effect measurements in CZ samples thermally treated at 470 °C [26] (see the introduction), which could well be associated with C_i–O_2 complexes, would accomplish within a few meV (0.74 versus 0.767 eV) the experimental value of the transition associated with the P line luminescence.

In this case the photoluminescence emission at 0.767 eV would present the isotopic carbon shift and its time evolution would closely follow that of TD.

This hypothesis is supported by the experimental evidence (see figure 12) of a different photoluminescence quenching and then, of a different TD decomposition kinetics in p-type and n-type samples, which should be associated with the well recognized role of boron in enhancing the precipitation of silicon oxide [28].

We suggest therefore that the decrease of the photoluminescence intensity observed after about 25 hours is due to a process of decomposition of the defect with the simultaneous generation of the embryos of the silicon oxide phase and ejection of self-interstitials [16]. We suggest as well that also the P line emission could be associated with the presence of over-coordinated oxygen, if the deep centre at $E_v + 0.35$ eV is a gap state associated with TDs.

5. Conclusions

Our systematic PL measurements support strongly the hypothesis, based on theoretical considerations about the structure of the interfaces between metastable oxygen clusters or oxide particles embedded in a crystalline silicon matrix, that over-coordinated oxygen and/or self-interstitials are intimately correlated with the generation of the centres which are responsible for the photoluminescence emission of dislocations, oxide precipitates and thermal donors in the 0.7–0.9 eV range.

A definitive conclusion requires however the determination by DLTS measurements of the energy of the deep level involved in the radiative recombination at TDs, in order to confirm earlier Hall effect measurements. It requires as well some local cathodoluminescence measurements on oxide particles in their initial stage of growth after short nucleation annealing and conclusive photoluminescence measurements of the emission intensity as a function of the annealing time in carbon-rich CZ samples. All these experiments are currently in an advanced stage of progress.

Acknowledgments

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