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## Di-interstitial defect in silicon revisited

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Infrared spectroscopy was used to study the defect spectrum of Cz-Si samples following fast neutron irradiation. We mainly focus on the band at  $533\text{ cm}^{-1}$ , which disappears from the spectra at  $\sim 170^\circ\text{C}$ , exhibiting similar thermal stability with the Si-P6 electron paramagnetic resonance (EPR) spectrum previously correlated with the di-interstitial defect. The suggested structural model of this defect comprises of two self-interstitial atoms located symmetrically around a lattice site Si atom. The band anneals out following a first-order kinetics with an activation energy of  $0.88 \pm 0.3\text{ eV}$ . This value does not deviate considerably from previously quoted experimental and theoretical values for the di-interstitial defect. The present results indicate that the  $533\text{ cm}^{-1}$  IR band originates from the same structure as that of the Si-P6 EPR spectrum.

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### I. INTRODUCTION

A lot of processes of semiconductor materials are affected by point defects and/or doping in the lattice.<sup>1–11</sup> Investigating small self-interstitial clusters in Si is technologically important as they play fundamental role in many solid-state processes affecting the mechanical and electrical properties of the material. It is therefore necessary to control them as their formation can cause detrimental effects.

Small interstitial clusters attract the interest of the research community for technological and scientific reasons. First, for technological purposes as (besides {311} extended defects) small interstitial clusters can also cause or/and contribute<sup>12,13</sup> to anomalous transient enhanced phenomena of dopants in silicon. Second, for scientific purposes as the evolution of small interstitial clusters to {311} extended defects is not completely understood. Therefore, any further information on this issue would improve the understanding of the mechanisms involved in the relative processes. Previous studies explored the fundamental behavior of these defects, especially their structure, their stability, and transformations.<sup>14–16</sup> It has been suggested<sup>17</sup> in particular that the di-interstitial complexes may be the “building blocks” acting as precursors for the formation of the {311} defects. Spectroscopic investigations have made advances in the research of the di-interstitial defect but there are still unclear points and a definite picture for the defect has not been conclusively established. Electron paramagnetic resonance (EPR) measurements concluded<sup>18</sup> that an EPR center called Si-P6 originates from a defect that comprises two self-interstitials placed symmetrically a substitutional atom. It was found that the structure has a {100} symmetry. Actually, its g-tensor exhibits a  $C_2$  or  $C_{1h}$  symmetry with the symmetry axis along the  $\langle 100 \rangle$  direction at low temperatures (200 K), but the symmetry becomes  $D_{2d}$  at

300 K. The Si-P6 signal corresponds<sup>19</sup> to the positive charge state of the defect. Interestingly, Kim *et al.*<sup>20</sup> based on DFT calculations introduced a model for the di-interstitial defect which differs from Lee’s model in (i) the orientation of the dumbbell atoms and (ii) the location of the center atom. They related their model with the Si-P6 center. Additionally, the calculations by Kim *et al.*<sup>20</sup> account for a transition between a  $C_{1h}$  and a  $C_{2v}$  symmetry of the defect. Also the transition to  $D_{2d}$  symmetry at room temperature is satisfactorily explained<sup>20</sup> by considering the thermal averaging motion of the atoms of the defect. Other theoretical calculations<sup>21</sup> using density functional theory calculated that the stress-tensor B of the suggested Si-P6 model of the defect differs substantially from that deduced experimentally.<sup>18</sup> Additionally, they calculated that the di-interstitial defect diffuses very fast in the lattice at room temperature and is essentially undetected at these temperatures, in agreement with other theoretical results<sup>22</sup> for various di-interstitial structural forms. As a result, they have suggested<sup>21</sup> that the Si-P6 EPR signal could not originate from a di-interstitial complex, but either from a metastable version of it or from a larger interstitial complex. Obviously, the assignment<sup>18</sup> of the Si-P6 defect to the di-interstitial defect is not universally accepted, and more work is required.

Although extended defects like the {311} complexes can be detected and studied by structural techniques as for example transmission electron microscopy<sup>23</sup> (TEM), small interstitials clusters cannot be efficiently detected by structural techniques and their microscopic identification relies on spectroscopic techniques such as EPR, deep level transient spectroscopy (DLTS), photoluminescence (PL), and infrared absorption (IR). As mentioned above, the EPR technique has detected a Si-P6 signal which was assigned<sup>18</sup> to the di-interstitial defect. A DLTS level at  $E_v + 0.40\text{ eV}$  observed<sup>24</sup> in neutron irradiated Si has similar thermal stability with the Si-P6 defect. Two other DLTS levels at  $E_c - 0.07\text{ eV}$  and  $E_c - 0.49\text{ eV}$  have also been correlated<sup>25</sup> with the di-interstitial defect. The well-known W photoluminescence band and at

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1.018 eV has also been suggested<sup>26,27</sup> to be linked with a certain compact configuration of the di-interstitial cluster, although other studies<sup>28</sup> have connected the W band with a certain configuration of the tri-interstitial defect.

The present work is focused on the study of the  $533\text{ cm}^{-1}$  IR band in neutron-irradiated Si. To this end, the thermal evolution of the  $533\text{ cm}^{-1}$  band was monitored carefully and its annealing kinetics was studied. The present results enable us to suggest that the  $533\text{ cm}^{-1}$  IR band and the Si-P6 EPR signal arise from the same defect that is a di-interstitial structure where two self-interstitials in the form of a dumbbell are centered on a lattice site Si atom.

## II. EXPERIMENTAL METHOD

We used three groups of *p*-type ( $[B] = 2 \times 10^{15}\text{ cm}^{-3}$ ) samples labeled  $S_1$ ,  $S_2$ , and  $S_3$  of 2 mm thickness with the following initial concentrations of the oxygen and carbon impurities:  $S_1$  ( $[O_i]_0 = 8.7 \times 10^{17}\text{ cm}^{-3}$ ,  $[C_s]_0 = 2 \times 10^{16}\text{ cm}^{-3}$ ),  $S_2$  ( $[O_i]_0 = 7 \times 10^{17}\text{ cm}^{-3}$ ,  $[C_s]_0 = 5.05 \times 10^{16}\text{ cm}^{-3}$ ), and  $S_3$  ( $[O_i]_0 = 6 \times 10^{17}\text{ cm}^{-3}$ ,  $[C_s]_0 < 10^{16}\text{ cm}^{-3}$ ). The samples were irradiated with 5 MeV fast neutrons at a fluence of  $1 \times 10^{17}\text{ n cm}^{-2}$ . The temperature of irradiation was lower than  $50^\circ\text{C}$ . After the irradiation, the samples were subjected to 20 min isochronal anneals, in steps of  $\Delta T \approx 10^\circ\text{C}$ . After each annealing step, IR spectra were recorded at room temperature by means of a JASCO-IR 700 spectrometer of dispersive kind. Intrinsic absorption was always subtracted by using a reference sample of float-zone Si of equal thickness.

## III. RESULTS AND DISCUSSION

Most of the results concerning radiation defects have been gathered from electron irradiations and from ion implantations. This is because electron irradiations and low fluence ion implantations produce simple point defects and therefore are advantageous for studies of simple defects. Conversely, neutron irradiation produces more complex defects. In the latter case, the spectra are more complicated. In general, relatively less experimental work has been done on neutron-irradiated Si. Neutron damage has some inherent characteristics. It generates<sup>29</sup> higher concentration of primary defects and the spatial separation<sup>30</sup> of the vacancies and the self-interstitials in the bulk favors the formation of larger clusters of these defects facilitating their investigations. It has been argued<sup>31</sup> that in neutron-irradiated Si almost 99% of the self-interstitials are annihilated through recombination with vacancies. In an intrinsic material irradiated by neutrons at a fluence of  $10^{18}\text{ cm}^{-2}$ , only about  $10^{16}\text{ cm}^{-3}$  self-interstitials would be available<sup>19</sup> for the formation of self-interstitial clusters and therefore for fluences about  $10^{17}\text{ cm}^{-2}$ , as in our case, one would expect about  $10^{15}\text{ cm}^{-3}$  self-interstitials. Obviously in our samples the respective signals, if any, from such clusters are expected to be very weak. In this context, we chose to present in Fig. 1 the IR spectra of samples from the  $S_2$  group, just for comparison purposes, in order to have a measure of the intensities of the detected bands along with already known weak bands of carbon and oxygen related defects. Carbon is a very important impurity in Si interacting readily<sup>32</sup> with many defects

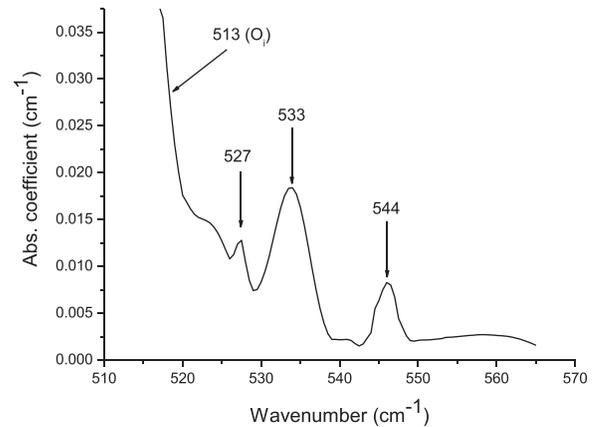


FIG. 1. Segment of the IR spectrum of the neutron-irradiated  $S_2$  sample.

and impurities present in the lattice, especially with self-interstitials, and can provide further insights. A weak band appearing in our spectra at  $527\text{ cm}^{-1}$  has been previously investigated<sup>33</sup> and correlated with the  $C_sC_s$  defect. Another weak band at  $544\text{ cm}^{-1}$  has also been studied<sup>34</sup> previously. It is a complex one arising from the contribution of two defects that is the  $C_iC_s$  and  $C_iO_i$  pairs having individual bands at  $543.5$  and  $545.5\text{ cm}^{-1}$ , correspondingly. It is worth noting that the  $527$  and  $544\text{ cm}^{-1}$  bands are not present in the spectra of the  $S_3$  samples where the carbon content is practically negligible and only traces of them could be detected in the spectra of the  $S_1$  samples which have low carbon content. As we mentioned above, this work will be primarily focused on the investigation of the  $533\text{ cm}^{-1}$  band.

Furthermore, we are concerned with the study of the thermal stability of the defects. In Fig. 2, the intensity of the  $533\text{ cm}^{-1}$  band is plotted as a function of the annealing temperature for the three samples. In the course of the 20 min isochronal annealing sequence from  $50^\circ\text{C}$  upwards, the band appears to be stable up to  $\sim 170^\circ\text{C}$ , where it begins to decay and eventually disappears at  $\sim 200^\circ\text{C}$ . It has the same thermal behavior as that of the Si-P6 EPR signal. To quantify the annealing process, we have modeled the data and concluded that they follow a first-order kinetics described by the rate equation:<sup>10</sup>

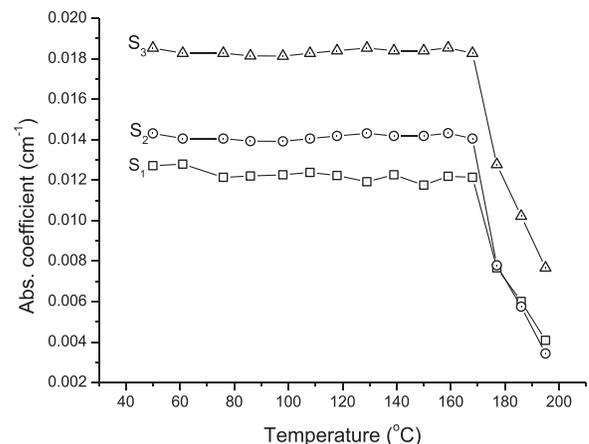


FIG. 2. The thermal evolution of the  $533\text{ cm}^{-1}$  band of the  $S_1$  (a), the  $S_2$  (b), and the  $S_3$  (c) samples.

$$\frac{dN}{dt} = -KN, \quad (1)$$

where  $N$  is the concentration of the defect and  $K$  the rate constant. By integrating, we get the expression

$$K\Delta t = \ln \frac{N_t}{N_{t+\Delta t}}, \quad (2)$$

where  $N_t$  is the concentration of the defect after annealing time  $t$  at a temperature  $T$  and  $N_{t+\Delta t}$  the concentration after annealing time  $t + \Delta t$ . Equation (2) provides values of the rate constant  $K$  for each annealing temperature  $T$ . By plotting  $\ln(K\Delta t)$  versus  $1/T$  (Fig. 3), it is observed that  $K\Delta t$  exhibits an Arrhenius behavior, that is  $K \propto \exp(-E_a/k_B T)$ , where  $E_a$  is the activation energy that characterizes the process and  $k_B$  is Boltzmann's constant.<sup>10</sup> The values of the activation energy deduced from the above analysis were  $0.87 \pm 0.02$  eV for the sample  $S_1$ ,  $0.88 \pm 0.03$  eV for the sample  $S_2$ , and  $0.89 \pm 0.04$  eV for the sample  $S_3$ . Thus, a mean value of 0.88 eV is derived with an error of  $\pm 0.03$  eV. Apparently, this value does not coincide with the experimental value of  $0.6 \text{ eV} \pm 0.2 \text{ eV}$  of the Si-P6 EPR center, extracted<sup>18</sup> from the response of the defect to external uniaxial stress. However, it is not unusual in the literature that reported values of a defect energies derived from different experimental techniques to deviate by 0.1–0.2 eV or even more.<sup>35</sup> In that respect, the above two values are not considered as inconsistent. In this sense, the two signals, namely the Si-P6 from EPR measurements and the  $533 \text{ cm}^{-1}$  band from IR measurements could be linked as originated from the di-interstitial defect. Notably, theoretical calculations have predicted a range of migration energies for the di-interstitials. These theoretical works suggest different structures for the di-interstitials and different diffusion mechanisms resulting in different diffusion barriers.<sup>20,21,36</sup> Consequently, a unanimously accepted structural model for the defect and its diffusion mechanism has not been reached. Concerning activation energies, values ranging from 0.5 to 0.89 eV have been reported in the literature.<sup>20,21,36</sup>

One step further to support the above correlation is to exclude possibilities that other radiation induced defects formed during irradiation of our material to be the origin of

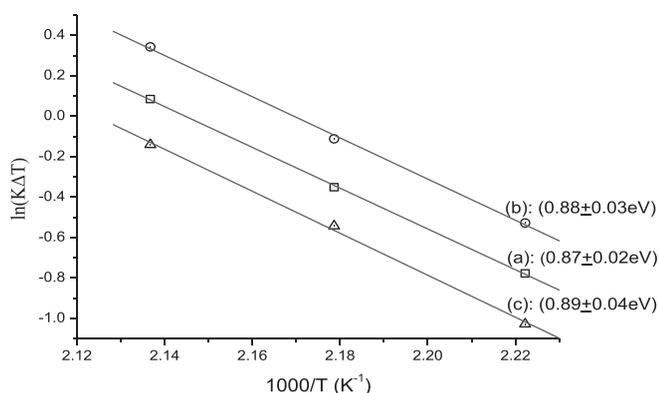


FIG. 3. Arrhenius plot for the decay of the  $533 \text{ cm}^{-1}$  band for the  $S_1$  (a),  $S_2$  (b), and the  $S_3$ (c) samples.

the  $533 \text{ cm}^{-1}$  band. Notably, in  $p$ -type (boron-doped) Cz-Si, as in the samples of our experiments, the  $\text{B}_i\text{O}_i$  defect is also expected to form upon irradiation. Significantly enough, there are no vibrations bands linked with the defect but it gives rise<sup>37</sup> to a deep level in the gap at  $E_c - 0.23 \text{ eV}$ . The corresponding DLTS peak disappears from the spectra in the temperature range  $150\text{--}200^\circ\text{C}$  showing similarities in its thermal stability with the  $533 \text{ cm}^{-1}$  band. However, its 20 min isochronal annealing curve depicted in Fig. 3 of the study of Kimerling *et al.*<sup>37</sup> is different from the corresponding one of the  $533 \text{ cm}^{-1}$  band. Interestingly, the  $E_c - 0.23 \text{ eV}$  peak begins to decay at  $\sim 150^\circ\text{C}$  although the  $533 \text{ cm}^{-1}$  band at  $\sim 170^\circ\text{C}$ . Furthermore, in electrical measurements the decay of the  $\text{B}_i\text{O}_i$  peak in carbon-rich Si is accompanied in the spectra by another peak ( $E_v + 0.29 \text{ eV}$ ) emerging at  $\sim 150^\circ\text{C}$  and attributed to the  $\text{B}_i\text{C}_s$  center. In our IR spectra of the  $S_3$  samples (not shown here), which contains adequate carbon there is no evidence of the emergence of any band when  $533 \text{ cm}^{-1}$  anneals out. Moreover, the activation energy of the  $\text{B}_i\text{O}_i$  is 1.2 eV much larger than the value of 0.6 eV of the Si-P6 defect. Another point: it is well-known<sup>31,38–40</sup> that carbon interacts strongly with self-interstitials. The fact that the  $533 \text{ cm}^{-1}$  band appears in all the samples used in this work, and in particular the  $S_3$  sample with carbon concentration below detection limit is an indication that the band is not directly related with the carbon impurity.

All these indications lead us to suggest that the  $533 \text{ cm}^{-1}$  IR band and the Si-P6 EPR spectrum in Si originate from the same center attributed to the di-interstitial defect. Our assignment was mainly based on the similarity of the thermal stability of the two centers the consistency between the extracted experimental values of the activation energies and the exclusion of other possible radiation defects that were candidates for the  $533 \text{ cm}^{-1}$  IR band.

## IV. CONCLUSIONS

This work was mainly focused on the study of a band at  $533 \text{ cm}^{-1}$  observed in the IR spectra of neutron-irradiated silicon. The band begins to decay  $\sim 170^\circ\text{C}$  exhibiting thermal stability similar to that of the Si-P6 EPR spectrum previously attributed to a di-interstitial defect. Its activation energy derived from IR measurements was found  $0.88 \pm 0.03 \text{ eV}$ . This value deviates from the value of  $0.6 \pm 0.2 \text{ eV}$  of the Si-P6 defect derived from EPR measurements, but the differences of 0.1–0.2 eV can be traced in the errors/assumptions of the two methodologies. Our analysis of the results has connected the  $533 \text{ cm}^{-1}$  with the di-interstitial defect giving rise to the Si-P6 EPR signal. Calculation of the vibrational frequency of the defect structure suggested from EPR measurements would provide additional support of the above attribution. Given that the information about di-interstitials, besides the huge volume of work is still meager and incomplete, it is expected that the present study will stimulate further investigations. This is also important from a technological point of view since agglomeration processes of self-interstitials in heat-treated Si are thought to form extended structural defects, which impact properties.

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