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Localised vibrational mode spectroscopy studies of self-interstitial clusters in neutron irradiated silicon

C. A. Londos,[1,a)] G. Antonaras,[1] and A. Chroneos[2,3]
[1]University of Athens, Solid State Physics Section, Panepistimiopolis Zografos, Athens 157 84, Greece
[2]Materials Engineering, The Open University, Milton Keynes MK7 6AA, United Kingdom
[3]Department of Materials, Imperial College, London SW7 2AZ, United Kingdom

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The evolution of self-interstitial clusters in silicon (Si), produced by fast neutron irradiation of silicon crystals followed by anneals up to 750 °C, is investigated using localised vibrational mode spectroscopy. A band at 582 cm^{-1} appears after irradiation and is stable up to 550 °C was attributed to small self-interstitial clusters (I_n, $n \leq 4$), with the most probable candidate the I_4 structure. Two bands at 713 and 758 cm^{-1} arising in the spectra upon annealing of the 582 cm^{-1} band and surviving up to ~750 °C were correlated with larger interstitial clusters (I_n, $5 \leq n \leq 8$), with the most probable candidate the I_8 structure or/and with chainlike defects which are precursors of the {311} extended defects. The results illustrate the presence of different interstitial clusters I_n, at the various temperature intervals of the material, in the course of an isochronal anneal sequence. As the annealing temperature increases, they evolve from first-order structures with a small number of self-interstitials (I_n, $n \leq 4$) for the temperatures $50 < T < 550$ °C, to second order structures (I_n, $5 \leq n \leq 8$) with a larger number of interstitials, for the temperatures $550 < T < 750$ °C. © *2013 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4816101]

I. INTRODUCTION

Si is a mainstream material with many applications in microelectronic, photovoltaic, and sensor devices. Importantly, the detailed understanding of many defect-dopant interactions affecting its properties are not well established.[1–5] Dopant-defect interactions become increasingly important as the characteristic dimensions of devices nowadays are a few nanometers or in the space radiation environment where a supersaturation of point defects is expected. In device fabrication processes, intrinsic defects always appear and play a crucial role in the behaviour of the corresponding devices. It is common knowledge, that self-interstitials form clusters in bulk Si. Their presence affects significantly the properties of Si, and, therefore, their study is necessary both from the fundamental and the technological point of view.

Self-interstitial clusters can be produced by neutron,[6,7] electron,[8] proton,[9] and alpha[10] particle irradiations as well as by ion implantations[11,12] of Si. Supersaturation of interstitials leads to the formation of {311} extended defects. {311} defects and small interstitial clusters are responsible[13,14] for transient enhanced diffusion (TED) of dopants in Si. TED place limitations in the scaling down of device sizes. Therefore, as the fabrication of submicron devices is very important for microelectronics, the understanding and control of interstitial defects has become a critical issue and the interest on these defects is very high.

It is established that {311} defects form by aggregation of small interstitial clusters. Noticeably, the {311} defects are detected by transmission electron microscopy (TEM),[15] and the experimental results are supported by theoretical studies[16] that have provided a clear picture for their structure. However, this is not the case for small interstitial clusters. Such defects of nanometer-size cannot be detected by structural techniques and their microscopic identification mostly relies on spectroscopic techniques as electron paramagneric resonance (EPR), deep level transient spectroscopy (DLTS), photoluminescence (PL), and infrared absorption (IR). The relative picture concerning the structure and the properties of the small interstitial clusters is far from being complete. Most importantly, the understanding of their behaviour under annealing and how they evolve to larger clusters and finally to {311} defects is still poor.

An AA12 EPR center has been correlated[17] with the single self-interstitial (I_1) in Si trapped at a tetrahedral site. The defect is annealed in the range of 280 to ~350 °K. A P6 EPR signal has been attributed[1] to the positive charge state of the di-interstitial (I_2) defect in Si. Nevertheless, theoretical calculations[18,19] indicate that the P6 EPR signal may arise from a larger cluster.

A W photoluminescent (PL) center with a zero-phonon line at 1.0182 eV is generally accepted to arise from self-interstitial aggregates. It has been proposed[20] to originate from the tri-interstitial (I_3) cluster. Its structure consists[20] of a three member ring of Si interstitials centered near the tetrahedral interstitial site. However, the compact structure of I_3 does not give rise[21] to the local modes associated with the W centre and another configuration of three self-interstitials may be the correct structure. It has been suggested[19] that tri-interstitial clusters could form at RT when mobile di-interstitials, as those suggested by Lee,[6] combine with a self interstitial. A B_5 EPR signal has also been correlated[7] with the tri-interstitial cluster. The defect appears after irradiation, increases its intensity between 200 and 300 °C, and then decreases and disappears[22,23] from the spectra in the range 400 to 500 °C.

a)electronic mail: hlontos@phys.uoa.gr

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Around 200 °C another center appears in the spectra, the X photoluminescent center with a zero-phonon line at 1.0398 eV. Its intensity increases up to 450 °C and then decreases, disappearing[22,23] from the spectra in the range 550 to 600 °C. The X center, as well as the W center, forms in most types of irradiation independently[24,25] of the carbon and oxygen doping. It has been clarified that it is an intrinsic center. The intensity of the 1.0398 eV line appears[25] to increase when the intensity of the 1.0182 eV line decreases, indicating that the X center is developed at the cost of the W center. The X center has been attributed[26] to the tetra-interstitial (I_4) cluster. Its structure consists[26] of four $\langle 001 \rangle$ split interstitial pairs which replace four next-nearest neighbour atoms lying in a common {001} plane. The positive charge state of the defect give rise[7,26] to the B_3 EPR center. Another EPR signal, NL51, observed under illumination, originates[27,28] from the neutral excitonic state of the center. A deep level at $E_v + 0.29$ eV has also been linked[26,29] with the defect. Theoretical calculations indicate that the tetra-interstitial cluster could form[30] either with the aggregation of a tri-interstitial and a single interstitial or by a combination of two di-interstitials. Interestingly, the tetra-interstitial can be formed[19] at the same time as the tri-interstitial.

Previous studies supported by theory on small interstitial clusters accumulated useful information on the structure and dynamics of these defects; however, little is known and understood on how they nucleate and evolve to form extended defects. As the number of self-interstitials increases, the thermal stability of the corresponding clusters also increases. The (X PL, B_3 EPR) signals of the I_4 defect anneal at a higher temperature than the (W PL, B_5 EPR) signals of the I_3 defect which anneals at a higher temperature than the P_6 EPR signal of the I_2 defect. It is reasonable to consider that the small interstitial clusters I_n ($n \leq 4$) could provide a nucleus for the formation of extended defects under annealing at high temperatures. Remarkably, the I_2,[6,18] the I_3,[31,32] and the I_4[26,33,34] clusters have been considered as the building blocks for larger clusters and extended defects. Various scenarios have been developed to account for the evolution of small I_n clusters to extended defects. In one scenario,[35] a sequential kinetic model for the growth of I_n clusters undergoing Ostwald ripening was suggested and two clusters I_4 and I_8 were found particularly stable. In this evolution scheme, the I_4 structure suggested by Arai *et al.*[33] is considered as the building block for the larger stable interstitial clusters. I_n clusters with $n \geq 15$ are found very stable and were considered to correspond to extended {311} defects. In another scenario,[36] it was suggested that with increasing number of self-interstitials in the cluster, the initially small clusters evolve from compact to interstitial chains and then to rod-like structures. The interstitial chains provide the basic building blocks of the extended {311} defects. Bongiorno *et al.*[37] proposed an evolutionary path from compact I_n ($n \leq 4$) clusters to elongated I_n ($n \geq 5$) clusters. Notably, experimental studies[38] strongly indicate that the small I_n clusters are not the direct precursors of the {311} defects and a severe structural transformation occurs in the evolution process from small I_n clusters to {311} defects. In any case, information on small interstitial clusters regarding their growth, shape, structure, energetics, and aggregation to various morphologies attract high interest and a combination of models and computation approaches[39–41] have been employed recently providing new insights on the issue.

This present study is focused on the investigation of LVM signals from self-interstitial clusters in neutron irradiated Si and the study of their evolution with temperature and their conversion from small to larger structures.

II. EXPERIMENTAL METHODOLOGY

p-Type boron-doped ($[B_s] = 2.6 \times 10^{15}$ cm^{-3}) Cz-Si prepolished samples of 2 mm thickness (from MEMC), with $[O_i]_o = 8.6 \times 10^{17}$ cm^{-3} and $[C_s]_o = 4 \times 10^{16}$ cm^{-3} were used. The samples were irradiated with fast neutrons with fluence of $D = 1 \times 10^{17}$ n/cm^2, at T = 40–50 °C. Neutron damage has some inherent characteristics. It generates[42] higher concentration of primary defects and the spatial separation[43] of vacancies and self-interstitials in the bulk favours the formation of larger clusters of these defects facilitating their investigation. Knowing the effect[44–46] of carbon on the self-interstitials and its tendency to readily capture them, we used samples with the lowest available carbon content to minimize its impact on the formation of self-interstitial clusters, which are the focus of the present work. Prior to irradiations, some of the samples were subjected to heat treatments at 1000 °C for 5 h. Material processing often necessitates thermal treatments at elevated temperatures. Such treatments also affect the general behaviour of I_n clusters, and the exact knowledge of their influence is significant for manufacturing Si-based devices. After the irradiations, the samples were subjected to isochronal anneals of ∼10 °C steps in an open tube furnace at temperatures ranging from 60 to 750 °C. In order to investigate the evolution of the radiation-induced defects, the IR spectra of the samples were carefully monitored after each annealing step. Absorption spectra were taken with a JASCO-700 IR spectrometer of dispersive kind. The background two-phonon absorption was always subtracted by using as reference a Float-zone sample of equal thickness with that of the Cz samples.

III. RESULTS AND DISCUSSION

Fig. 1(a) shows a section of the IR spectra of the as-grown samples. The spectra were recorded after the 77 °C annealing stage of the isochronal sequence. Some of the bands have already been identified. For example, the 527 cm^{-1} band has been attributed[47] to the C_sC_s defect. The 533 cm^{-1} band has been argued,[48] on the basis of its thermal stability, that has the same origin as the Si-P6 EPR spectrum tentatively correlated[6] with the Si di-interstitial. The band at 544 cm^{-1} is a complex one arising[48] from the correlation of two bands at 543.5 and 545.5 cm^{-1} attributed to the C_iC_s and C_iO_i defects, correspondingly. The 635 cm^{-1} band was also attributed[48] to the C_iC_s defect. The 604 cm^{-1} band is the well-known band of the C_s impurity, and the 667 cm^{-1} band is the optical signature of the CO_2. In what follows, we shall focus mainly on the 582 cm^{-1} band.

Fig. 1(b) shows the same section of the IR spectra as in Fig. 1(a) for the sample subjected to a heat treatment at 1000 °C for 5 h, prior to irradiation. The spectrum was

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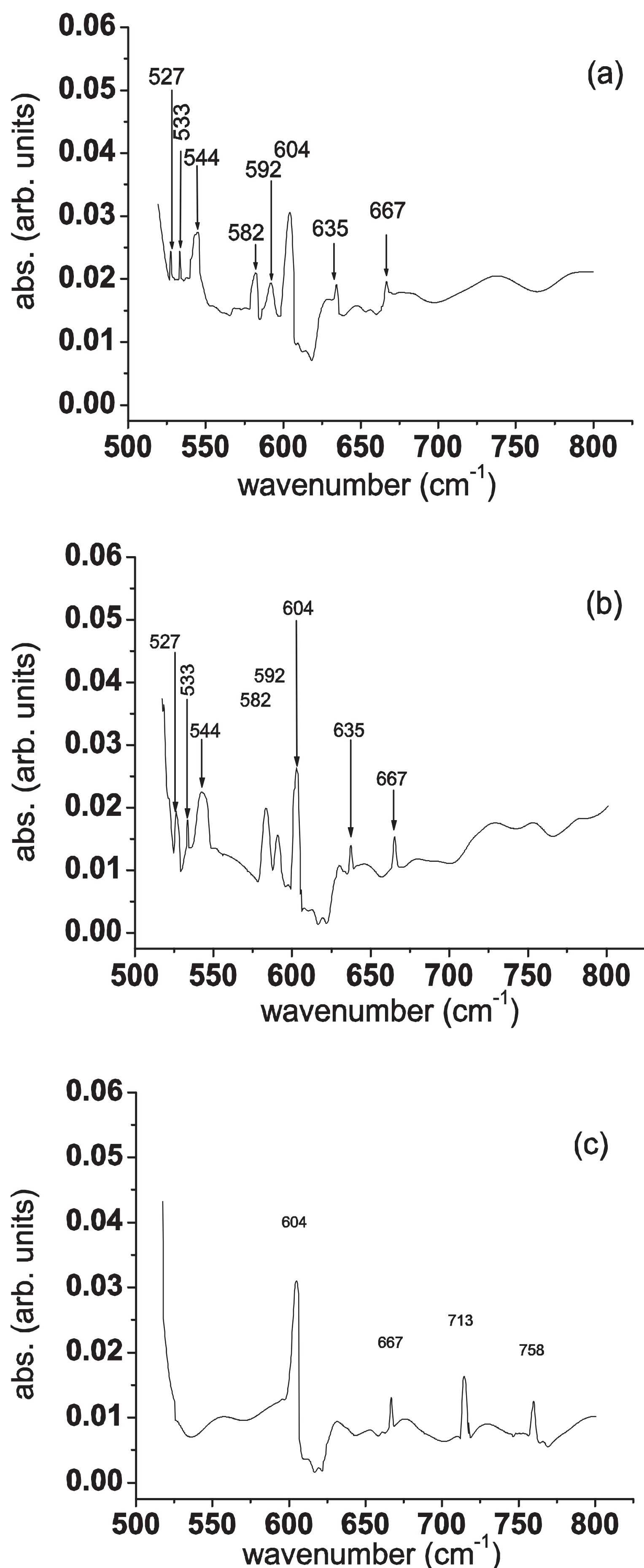

FIG. 1. (a) Section of the absorption spectra (range: 500–800 cm^{-1}) of the as-grown sample (recorded at T = 77 °C), (b) of the pre-treated sample at 1000 °C for 5 h (recorded at T = 77 °C), (c) of the pre-treated sample at 1000 °C for 5 h (recorded at 600 °C).

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recorded at 77 °C. Some of the IR bands in the spectra appear stronger in the samples subjected to heat treatments before the irradiation. Thus, the 604 cm^{-1} band of C_s and other carbon-related bands appear stronger. We have previously[48] argued that carbon atoms trapped at some complexes are liberated upon the thermal treatment, increasing the initial concentration of the carbon impurity and, therefore, the concentration of the carbon-related bands. Also carbon atoms emanated from precipitates contribute to the increase of the carbon-related bands. Similar arguments could be put forward for the bands related to interstitial clusters, as for example, the 533 cm^{-1} band which has been tentatively attributed[48] to a self-interstitial cluster and which appear stronger in the spectra of the initially heat-treated samples. In the latter samples, oxygen precipitates are expected to form. Their formation and growth is followed by the emission of self-interstitials for relieving the developed strains. Most of these self-interstitials are trapped at the precipitate/matrix boundary. Other self-interstitials may also be trapped at large defect clusters and other structural defects formed as a result of the thermal treatments. A number of these self-interstitials are liberated upon irradiation providing additional self-interstitial atoms, thus enhancing the intensity of the self-interstitial related bands. It means that the sinks of self-interstitials created as a result of the thermal treatment become, in the course of the irradiation, sources of self-interstitials. In other words, the thermal pre-treatments rearrange the availability of the self-interstitials, affecting their contribution in the various reaction channels they participate. Notably, the 582 cm^{-1} band appears stronger in the spectra of the pre-treated samples, as we can see by comparing Figs. 1(a) and 1(b). Fig. 1(c) represents the same section of the IR spectra recorded after the 600 °C annealing stage of the isochronal sequence and two new bands at 713 and 758 cm^{-1} are recorded. The thermal evolution of the 582 cm^{-1} band is shown in Fig. 2, together with the thermal evolution of the bands at 713 and 758 cm^{-1} arising in the spectra upon decaying of the 582 cm^{-1} band.

The 582 cm^{-1} band is stable up to 550 °C and then decays and disappears completely at 600 °C. At first, it is tempting to consider this band as originated from self-interstitial clusters, since it shows an annealing temperature well above 450 °C, where electrical signals from vacancy-related defects disappear.[49] The band shows a rather similar

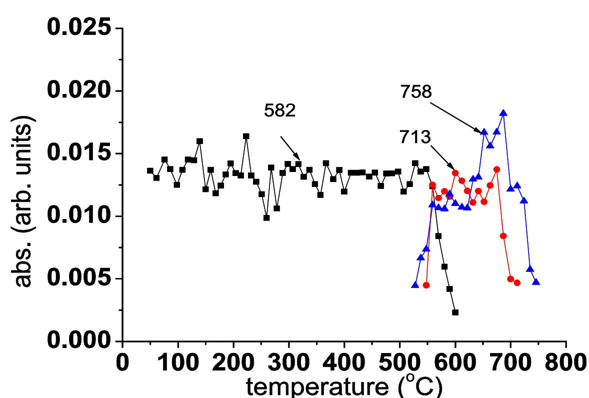

FIG. 2. The thermal evolution of the 582 cm^{-1} and the 713 and 758 cm^{-1} bands.

thermal stability with those of the B_3 EPR signal and the X PL center, which both originate[26,28] from the I_4 cluster. However, there are some differences between the annealing behaviour of the 582 cm^{-1} band and that of the (B_3 EPR, X PL) signals of I_4. The latter signals appear[22,23] in the corresponding spectra generally around 200 °C, show an increase up to 450 °C, and then decrease and disappear around 550 to 600 °C, although the former band appears in the spectra immediately after the irradiation, is almost stable up to 550 °C and then decreases and disappears completely at 600 °C. Theoretical investigations[19,50] have indicated that small interstitial clusters may possess more than one configurations, which are likely to coexist when they form, and some of these structures are very mobile and coalesce leading to the formation of tetra-interstitials, at room temperature. Such an option is particularly favoured in heavy dose neutron-irradiated Si, as in our case. Furthermore, considering[51] that the formation energy for the pairing of the interstitials is small, the number of available interstitials determine their growth. Thus, larger clusters are expected to form in the case of neutron irradiation. It was also found[51] that in implanted material, the stability of the clusters increase with higher doses. Extending the validity of this result for our heavily neutron irradiated Si, it may provide explanation for the surviving of the cluster responsible for the 582 cm^{-1} band to temperatures up to 600 °C. It deserves noting that two interstitial-related bands at 530 and 550 cm^{-1} previously reported[52] in neutron-irradiated Si, have been linked[26] with the B_3 EPR signal of the tetra-interstitial. Our 582 cm^{-1} band has annealing characteristics with the 530 and 550 cm^{-1} pair of bands, and they probably have similar origin. Furthermore, six electrical levels in *p*-type Si, at $E_v + 0.08$ eV, $E_v + 0.13$ eV, $E_v + 0.23$ eV, $E_v + 0.25$ eV, $E_v + 0.39$ eV, and $E_v + 0.53$ eV originated from self-interstitials clusters[53] have similar thermal stability (refer to Fig. 3 in Ref. 53) with our 582 cm^{-1} band. Interestingly, four sharp PL bands[11,12] at 997.5, 981.9, 972.1, and 965.3 meV may originate from the same range of small I_n clusters.

The 713 and 758 cm^{-1} bands have similar thermal stability with the 1.32 and 1.40 μm broad bands attributed[11,53] to second order interstitial clusters, although the authors of the above two reports disagree whether they originate from structures, which are direct precursors or not of the {311} extended defects. It has been proposed[54] that they may be related to either I_8 clusters or/and (100) interstitial chains. In a subsequent study,[55] the sharp PL bands observed in the range 1228–1400 nm, which is the same range where the bands at 997.5, 981.9, 972.1, and 965.3 meV are observed, have been attributed to big interstitial clusters such as the I_8 defects possessing multiple configurations and the two broad bands at 1.32 and 1.40 μm to chainlike structures. Cowern *et al.*[35] in the sequential formation model found that magic numbers for stable interstitial clusters are $n = 4$ and $n = 8$ and it seems likely that our 713 and 758 cm^{-1} bands to be linked with the I_8 cluster, as far as the 582 cm^{-1} band is linked with the tetra-interstitial. This assumption, however, does not rule out the possibility that the 713 and 578 cm^{-1} bands to be linked to chainlike defects[36,55] developed upon annealing, as the compact structures, represented by the 582 cm^{-1} band in our spectra, evolve to chainlike structures

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prior to becoming rod-like defects. It is worth noting that theoretical calculations show[56] that the leading mechanism for defect nucleation is not the I-chain configuration and more compact smaller structures are formed. Stable compact interstitial clusters with 8 members have been suggested.[35,57] These results were further supported by direct observation[58] of the enhanced stability of the I_8 cluster. Furthermore, atomistic simulations of the Ostwald ripening process for the nucleation and growth of the interstitial clusters show[59] that the transition from compact to elongated structures occurs above the cluster with "n = 8" members. Theoretical calculations also show[30] that beyond size 8, chainlike defects become energetically more favourable than compact defects.

IV. CONCLUSIONS

The investigation of the production of self-interstitial clusters in Si as well as their aggregation to larger morphologies by LVM spectroscopy in combination with theoretical and experimental results cited in the literature provides new elements of information in understanding the physics of these defects that play such an important role in semiconductor technology. Here, we present infrared spectroscopy studies of room temperature irradiated Si. The results unambiguously show the formation of I_n clusters. A band at 582 cm^{-1} appearing in the spectra immediately after the irradiation has been correlated with first order interstitial clusters (I_n, $n \leq 4$). In particular, we have tentatively linked the 582 cm^{-1} band to the I_4 structure. Two bands at 713 and 758 cm^{-1} appearing in the spectra upon annealing of the 582 cm^{-1} band around 550 °C have been linked with second order interstitial clusters (I_n, $5 \leq n \leq 8$), with most probable candidate either the I_8 structure or a chainlike defect.

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