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Production and evolution of defects in neutron-irradiated Si subjected to thermal pre-treatments under hydrostatic pressure

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Abstract

Thermal treatments of Czochralski-grown Si at $T = 450$, 600 and 650 °C, under high hydrostatic pressure of $P \approx 11$ kbar, introduce thermal donors and various structural defects, as for example oxygen precipitates. Neutron irradiation of such samples results first in the formation of oxygen–vacancy complexes, mostly VO defects. Upon annealing, the VO defects evolve in larger clusters via the accumulation of oxygen atoms and vacancies in the initial VO core, leading to the formation of V_mO_n defects. We focus on the study of the effect of pre-treatments on the production and evolution of the various V_mO_n defects upon isochronal annealing. The observed changes and variations in the IR spectra and the evolution curves in comparison with the corresponding ones of an initially untreated sample are discussed and some explanations are offered. The most important finding of this work is that the concentrations of the VO_2 and the VO_3 defects are reduced in the sample pre-treated at 450 °C, an indication of interaction between thermal donors and radiation-induced defects.

1. Introduction

The improvement of the functionality of the Si electronic components in various devices requires the control of the defects formed upon processing of the material. The more we know about the defects and their properties, the better we can improve the yield and the performance of the devices. In other words, advances in Si technology require deeper insight into the physics and the behaviour of the defects.

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Oxygen is the main, unintentionally added, impurity in Si crystals during growth. Its significance, besides the scientific interest, derives from the technological importance of Cz-grown Si, which is the basic material used as a substrate for the fabrication of integrated circuits. Two kinds of oxygen-related centres are normally observed in Si material. One kind of defects involves complexes formed due to irradiation which leads to the formation of centres containing primary defects (V, Si_I), for example the VO and the O_iSi_I centres, and also pairs with other impurities present in the material. In the case of neutron irradiation it is also possible that more than one vacancy can be trapped by an oxygen atom to form larger clusters. The other kind of defects involves centres formed due to thermal treatments. It is well-known that thermal treatments of the Si material trigger oxygen aggregation processes leading to the formation of thermal donors (TDs), oxygen precipitates and other structural defects.

The Si technology currently used by the industry involves thermal treatment steps of the material at elevated temperatures and also implantations and/or irradiations with various particles. All the above-mentioned processes introduce defects. From the practical point of view, it is therefore very interesting and important to study the basic interactions between the two categories of defects and also to study the impact of the presence of the one kind of defect on the behaviour and the properties of the other. Knowledge of the influence of the oxygen precipitates and accompanying structural defects upon the radiation-induced defects is very important in the case of Si materials being subjected to processes that involve both thermal treatments and irradiations.

Thermal treatments in the temperature range 450–650 °C mainly introduce various kinds of TDs [1]. Depending on the duration of the heat treatment with or without the application of high hydrostatic pressure small oxygen precipitates also form [2, 3]. Generally, in the temperature range of our interest in this work, ribbon-like precipitates are expected [4] to form. The effects of these defects in the formation and evolution of radiation-induced defects are still unresolved. There are conflicting reports in the literature. It has been reported [5] that thermal donors can be annihilated by irradiation, but also that primary defects produced by the irradiation do not interact [6] with thermal donors. The main objective of this study is to investigate the effect of the thermal defects, mainly thermal donors and/or small oxygen precipitates formed at the early stages of the oxygen aggregation process on the behaviour of radiation-induced defects in Si, mainly the V_mO_n defects. The exact knowledge of the thermal stability and generally the annealing behaviour of the oxygen–vacancy-related defects produced by irradiation in Si samples, pre-treated at various temperatures under high hydrostatic pressure, are expected to enable a better understanding of their mutual influence and interactions.

The spectroscopic investigation of the above issue, including the study of defect reactions, is substantially facilitated when the concentration of the involved defects increases. To this end, we have carried out neutron irradiations, and the thermal treatments were conducted with the application of high hydrostatic stress. First, it is well-known [7] that in neutron-irradiated material, due to the special separation of the primary defects, more vacancies are available to participate in secondary reactions and therefore the V_mO_n defects produced by the irradiation and the accompanied thermal treatments are expected to have higher concentrations than those, for example, in the electron-irradiated material. Second, it is known [8–10] that the application of stress increases the total concentration of TDs and enhances the oxygen precipitation process. This behaviour is believed [10] to be due to the enhancement of the diffusivity of single oxygen atoms and small oxygen aggregates. However, the application of stress induces [11–13] changes in the density and the structure of defects, which complicate the reactions. With the application of stress the same concentration of TDs is achieved in shorter time duration than that of the thermal treatment without stress. It may be argued, therefore, that we could have produced the same concentration of TDs by heat-treatments of longer duration and thus we could have avoided any complications involved by the application of external stress. However, high pressure is an external parameter, as is the temperature as well, and it is useful to do relative studies. More importantly, defect-engineering of oxygen in silicon wafers is not an isolated process. Other impurities and defects, as well as other parameters, i.e. external pressure, depending on the thermal treatment strategy, are of high importance, and therefore considerable attention has been directed in knowing the details of their influence.

2. Experimental details

In this work we used four Cz-grown Si material samples with initial oxygen concentration of $[O_i] \approx 1.4 \times 10^{18}$ cm^{-3}. The carbon substitutional concentration in our samples was beyond the IR detection limit ($[C_s] < 1 \times 10^{16}$ cm^{-3}). The as-received sample is labelled A_o, and the three samples subjected to high-temperature–high-pressure [14] (HTHP) treatments are labelled as follows: A_2 (450 °C, 10.7 kbar, 5 h), A_5 (600 °C, 12 kbar, 5 h), A_6 (650 °C, 11 kbar, 5 h). The short time duration of the anneal at these temperatures ensures that only thermal donors of various types and small oxygen precipitates form [2, 3]. Dislocation loops and/or stacking faults, if any, are considered to produce subtle effects. The small variations in the applied hydrostatic pressure (above 10 kbar) among the samples are considered of minor importance. Thus, pressure and time are considered constant and only the temperature of the treatment changes in the range 450–650 °C. The TD concentrations were extracted from Hall-effect measurements and were found to be 8×10^{15} cm^{-3} for sample A_2, and 2×10^{14} cm^{-3} for samples A_5 and A_6. After the heat-treatment steps, all samples were irradiated by fast neutrons at a fluence of 8×10^{16} n cm^{-2}. Post-irradiation annealing was carried out up to 600 °C in $\Delta T = 10$ °C and $\Delta t = 30$ min steps. The thermal evolution of the various defects was studied by monitoring the amplitudes of the corresponding peaks in the IR spectra, received after each annealing step, with the aid of a Jasco IR-700 spectrometer of dispersive kind.

3. Experimental results and discussion

The most important defect, besides the divacancy (V_2), formed in neutron-irradiated Cz-Si is the VO pair, the well-known A-centre, which in the neutral charge state gives rise [15] to an IR peak at ~828 cm^{-1}. Upon anneals [15, 16] at ~300 °C, the VO defect converts to the VO_2 defect (887 cm^{-1}), which in turn at ~450 °C converts to the VO_3 defect (903, 967 and 1004 cm^{-1}) and so on. A band at 983 cm^{-1} has been tentatively attributed [17] to a modified VO_3 defect with one more oxygen atom attached. The successive production of VO_n ($n = 1, 2, 3, \ldots$) defects take place via the successive aggregation of oxygen interstitial atoms on the initial formed VO defect. Along with this, another reaction channel leads to the formation of V_mO defects ($m = 1, 2, 3, \ldots$), via the successive accumulation of vacancies to the initial VO defects, while V_mO_n defects are also produced [16, 18] in the course of the annealing process. In a previous work [19], we have correlated the peaks at 839, 833, 824 cm^{-1} with the V_2O, the V_3O_2 and the V_2O_2 respectively. From their position in the spectra, these defects appear as satellite bands of the much stronger VO band. Note that, due to the pre-treatment steps, the samples are expected [20, 21] to contain thermal donors and small oxygen precipitates.

We shall first deal with the interaction, if any, between thermal and radiation-induced defects during the irradiation. As is seen from table 1, the amplitudes of the VO defects are about the same in samples A_o (as-grown) and A_2 (450 °C, 10.5 kbar, 5 h), indicating that no interaction occurs between VO and the TDs present in sample A_2. On the other hand, the amplitudes of the VO defects are considerably less in samples A_5 (600 °C, 12 kbar, 5 h) and A_6

Table 1. The [O_i] after treatment and after irradiation, as well as the VO amplitudes after irradiation.

Sample	O_i after treatment ($\times 10^{18}$ cm^{-3})	O_i after irr. ($\times 10^{18}$ cm^{-3})	VO (arb. units)
A_o (as received)	1.4	1.37	0.053
A_2 (450 °C, 10.5 kbar, 5 h)	1.38	1.28	0.052
A_5 (600 °C, 12 kbar, 5 h)	1.35	1.33	0.040
A_6 (650 °C, 10.9 kbar, 5 h)	1.4	1.35	0.045

(650 °C, 10.9 kbar, 5 h), compared to that of sample A_o. Notice that in the A_5 and A_6 samples, besides the presence of TDs, which are much less than in sample A_2, small oxygen precipitates are expected to form. As a result, Si_I are injected during the precipitation process, and some of them are trapped at the Si/SiO_x interface [20]. Thus, the decrease in the VO concentration could be attributed to the destruction of the VO defects by mobile Si_I liberated from the Si/SiO_x interface during irradiation ($VO + Si_I \rightarrow O_i$). This is supported by the fact that the difference ($[O_i]_{\text{after HTHP}}-[O_i]_{\text{after irr.}}$) is less in samples A_5 and A_6 than in the untreated sample A_o (see table 1), indicating that some of the oxygen atoms were restored during irradiation at interstitial sites, for pre-treatments at 600 and 650 °C.

We shall secondly deal with the interaction, if any, between thermal defects and radiation-induced defects during thermal anneals. Figure 1 depicts the evolution of the various V_mO_n defects in these samples. For comparison purposes the evolution of these defects in an as-received sample is also shown. As is seen from figure 1(a), the maximum intensity of the VO_2 defect in the 450 °C pre-treated sample is significantly lower than that in the as-received sample. This may be taken as an indication that the VO defect participates in additional reactions besides its normal conversion reaction to the VO_2 defect ($VO + O_i \rightarrow VO_2$). Because of the presence of thermal double donors (TDDs) an interaction between them and the VO defects cannot be excluded. Such an interaction has been proposed previously [22], although a direct experimental verification does not exist so far. As a consequence of the lower VO_2 concentration in the 450 °C pre-treated sample, the maximum intensities of the VO_3 LVM bands are also lower than those in the as-received samples. On the other hand, in the 600 and 650 °C pre-treated samples (figure 2(a)) the maximum intensity of the VO_2 signal is approximately equal to that of the as-received samples. It is well-known that heat treatments of short time duration at 650 °C destroy any TDDs present in the material due to the growth process. Moreover, longer time treatments or short time treatments coupled with high hydrostatic pressure (higher than 10 kbar) stimulate [3] the oxygen precipitation process together with the formation of another family of thermal donors, the so-called new thermal donors (NTDs), which have a different structure [21] than that of the TDDs. Our results indicate that any interaction between the VO defects and the thermal defects formed at 600 and 650 °C does not take place. However, it may be argued that since the concentration of the thermal defects induced upon treatments in the latter temperatures is not high enough, it is possible that any complex between them and the radiation defects is not detected in the IR spectra. It is also possible that any such complex that presumably forms may be IR inactive and therefore its presence remains undetected in the IR spectra. One way to resolve this issue is to monitor the intensity of the various defects formed during the isochronal annealing steps in the differently pre-treated samples and compare the results.

In table 2 the percentage of the VO defects converted to the VO_2 defects is given. Notice that in the A-centre the intensity of the 828 cm^{-1} band corresponds to the vibrations of the single oxygen atom of the defect. However, in the case of the VO_2 defect the intensity of the

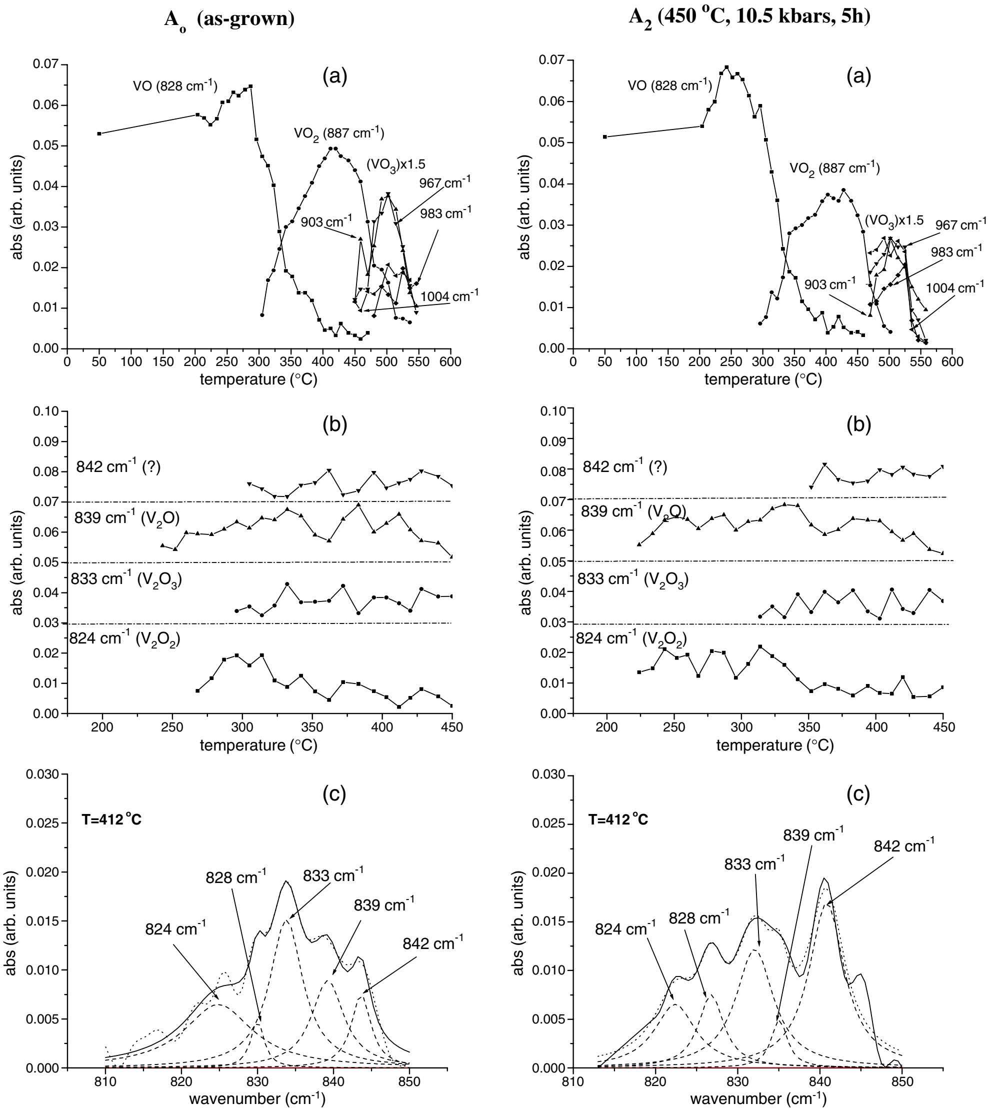

Figure 1. (a) The thermal evolution of the VO, VO_2 and VO_3 defects for the neutron-irradiated samples: A_o (as-received) and the HTHP pre-treated sample A_2 (450 °C, 10.5 kbar, 5 h). (b) The thermal evolution of the 839 cm^{-1} (V_2O), 833 cm^{-1} (V_3O_2), 824 cm^{-1} (V_2O_2) and 842 cm^{-1} bands of the A_o and A_2 samples. (Since the bands are very weak the corresponding peaks have been shifted upwards: the 833 cm^{-1} band has been shifted for 0.03 au, the 839 cm^{-1} band for 0.05 au and the 843 cm^{-1} for 0.07 au correspondingly) out of the necessity to have a clear picture of their evolution. (c) The deconvolution of the A-centre frequency range in the contributing bands, by using Lorentzian profiles, for the samples A_o and A_2.

887 cm^{-1} band corresponds to the contribution of the vibrations of the two oxygen atoms that share a common vacancy site and vibrate independently. This means that if all the VO centres were converted to the VO_2 defects the corresponding amplitude of the latter should be twice as that of the former. In other words, the calibration factor of the VO_2 defect is only half that of the VO defect. If N_{VO} and N_{VO_2} are the concentrations and α_{VO} and α_{VO_2} the calibration

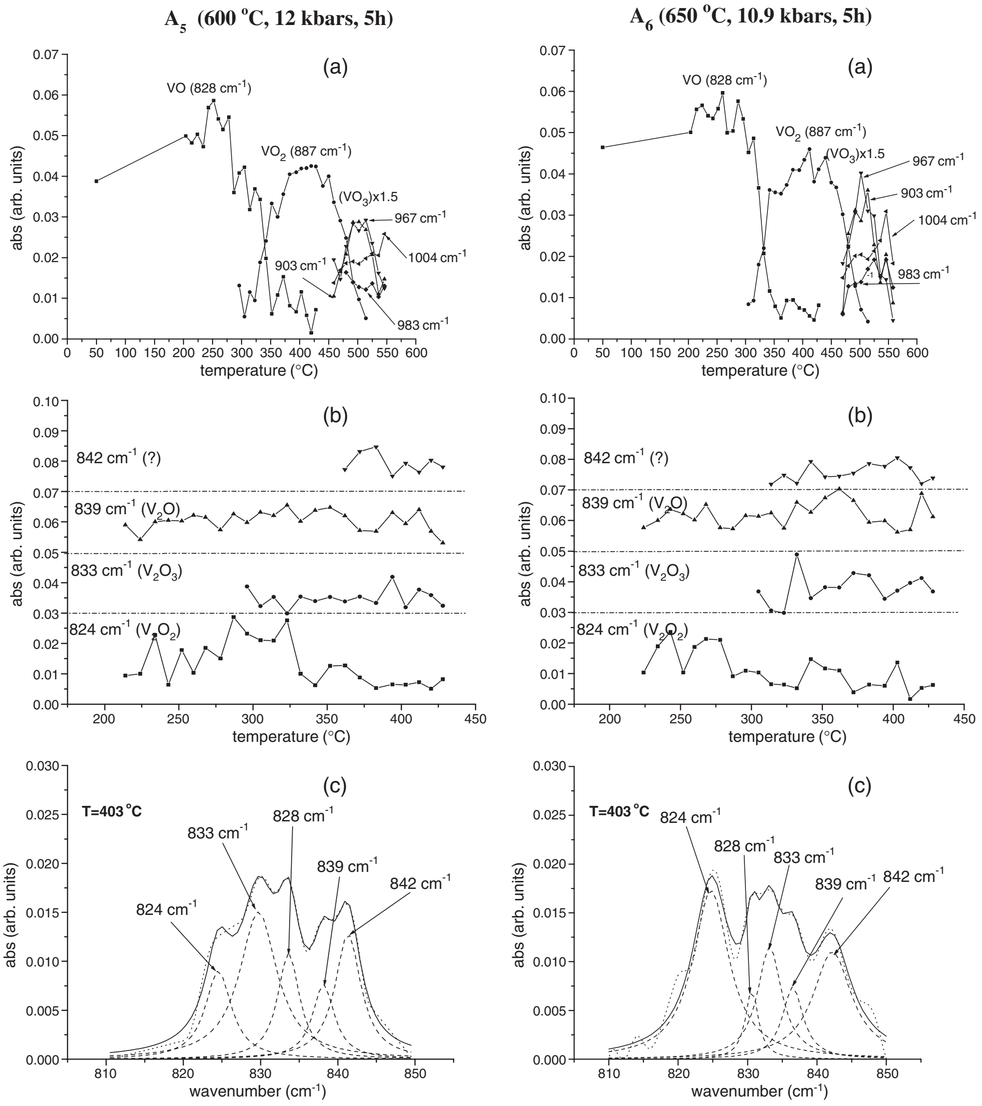

Figure 2. This figure is the corresponding of figure 1 for the HTHP pre-treated samples A_5 (600 °C, 12 kbar, 5 h) and A_6 (650 °C, 10.9 kbar, 5 h).

factors of the VO and the VO_2 defects respectively, then the concentration ratio of the two defects would be

$$\frac{N_{VO_2}}{N_{VO}} = \frac{h_{VO_2} \cdot a_{VO_2}}{h_{VO} \cdot a_{VO}} = \frac{h_{VO_2} \cdot a_{VO}/2}{h_{VO} \cdot a_{VO}} = \frac{1}{2}\frac{h_{VO_2}}{h_{VO}} \tag{1}$$

where h_{VO} and h_{VO_2} are the amplitudes of the VO and the VO_2 peaks in the spectra. In this approximation, the ratio of the integrated intensities of the two bands were taken equal to the ratio of the corresponding amplitudes in the spectra.

Thus, from the evolution curves presented in figures 1(a) and 2(a) it is concluded that the reaction channel $VO \xrightarrow{O_i} VO_2 \xrightarrow{O_i} VO_3$ is affected by the HP pre-treatment at 450 °C, where

Table 2. The maximum amplitudes of the VO and the VO_2 defects and the corresponding percentage of the VO → VO_2 conversion for the HTHP pre-treated Si samples.

Sample	VO max	VO_2 max	VO → VO_2 (%)
A_o (as-received)	0.0647	0.0493	38
A_2 (450 °C, 10.5 kbar, 5 h)	0.0683	0.0374	27.4
A_5 (600 °C, 12 kbar, 5 h)	0.0586	0.0425	36.3
A_6 (650 °C, 10.9 kbar, 5 h)	0.0596	0.046	38.6

thermal donors are present in the samples. The evolution curves of the VO_2 and VO_3 defects for the 600 and 650 °C HP pre-treated samples have no significant changes in comparison with the untreated sample. The argument presented above about an interaction between TDs and the VO defect holding for the sample pre-treated at 450 °C, but not for the samples pre-treated at 600 and 650 °C, is further supported by the fact that in the former case the sample A_2 has much higher concentration of TDs (8×10^{15} cm^{-3}) than that in the latter case of the samples A_5 and A_6 where the TDs concentration was 2×10^{14} cm^{-3}. On the other hand the intensities and generally the evolution of the V_2O, V_2O_2, V_3O_2 bands (figures 1(b) and 2(b)) are approximately the same in all samples, indicating that these defects seem not to be affected by the pre-treatments at 450, 600 and 650 °C.

By inspection of the evolution curves of the VO defect (figures 1(a) and 2(a)) of our samples, it is seen that the annealing temperature of the defect is lower in samples A_2, A_5 and A_6, pre-treated under HP than that of the untreated sample A_o. Notice that two main reactions participate [23, 24] in the annealing process of the VO defect, that is $VO + O_i \rightarrow VO_2$ and $VO + Si_i \rightarrow O_i$. In the case of the untreated sample A_o, sources of the Si_I are considered [25] large defect clusters. In the case of the pre-treated samples A_5 and A_6 where precipitates form there is an additional source of Si at the Si/SiO_x interface. In the sample A_2 pre-treated at 450 °C under HP, some small precipitates are also expected to form. It has been reported [26] that pre-heat treatments at 600 °C cause a decrease in the annealing temperature of the VO defect. The phenomenon was attributed to the agglomeration of Si_I at the Si/SiO_x interface, which upon liberation lead to the destruction of the VO defect according to the reaction $VO + Si_I \rightarrow O_i$. We argue, therefore, that in our case the annealing temperature of the VO defect occurs in the HP pre-treated samples at a lower temperature, due to the liberation of the trapped Si_I from the Si/SiO_x interface. Understandably, the reaction $VO + Si_i \rightarrow O_i$ is triggered earlier than that in the initially untreated sample, resulting in the decay of the VO band in the spectra at a lower temperature.

Figures 1(c) and 2(c) show computer deconvolution, by using Lorentzian profiles, of the frequency range in the spectra around the A-centre band, for temperatures above 400 °C. In doing Lorentzian profiles, we have allowed for different values of the half widths in the deconvolution process of the VO band so that the best fit of the corresponding IR spectrum is obtained. As a result, we have obtained different values of the half widths of the same bands in different samples. However, the relative variation of the half widths was not so large as to affect the peak amplitudes of the bands significantly. As is seen from figures 1(c) and 2(c), the total A-centre band contains the contribution of four individual bands. As mentioned above, three of them, that is the 839 cm^{-1} band (V_2O), the 833 cm^{-1} band (V_3O_2) and the 824 cm^{-1} band (V_2O_2), have been previously detected [19] and studied. In this work, detailed Lorentzian analysis revealed the presence of an additional satellite band at 842 cm^{-1}. Its thermal evolution is also presented in figures 1(b) and 2(b). As is seen, the band appears around ∼350 °C and persists in the spectra up to ∼450 °C. The defect is present in the spectra in a temperature range which is approximately the same with that previously reported [27], from EPR studies, for the V_3O_3 defect. On the other hand, recent measurements [28] making use of positron lifetime and coincidence Doppler broadening techniques have concluded that when VO centres anneal out they lead to the formation of more stable larger complexes, the V_3O and the V_4O_2 defects. However, the V_3O defect has been correlated [29] with another band at 887 cm^{-1}. Notice, in addition, that multivacancy–multioxygen centres, such as the V_3O_3 and the V_4O_2 structures, are expected to form in heavily irradiated material. Thus, we tentatively correlate the 842 cm^{-1} band with either the V_3O_3 or the V_4O_2 structure in neutron-irradiated silicon. More work is needed in order to establish a more definite attribution.

4. Conclusions

The effect of the HTHP pre-treatments on the production and evolution of the radiation-induced oxygen-related defects in Cz-grown Si material was investigated. During irradiation no interaction between thermal donors and VO defects was found, although an indirect interaction between oxygen precipitates and VO defects was inferred. During thermal anneals the reaction channel $VO \xrightarrow{O_i} VO_2 \xrightarrow{O_i} VO_3$ was found to be affected by the HP pre-treatments at 450 °C. In that case, the concentrations of the VO_2 and the VO_3 defects are lower than that of the as-received material. The phenomenon was tentatively attributed to the interaction between thermal donors and VO centres. Interestingly enough, the concentrations of the VO_2 and VO_3 defects were not affected by the HTHP pre-treatments at 600 and 650 °C, where other kinds of donors and thermal defects are produced, which apparently do not interact with the VO centres. Additionally, we found that the thermal evolution of the V_2O, V_2O_2 and V_3O_2 defects is not affected by any of the above pre-treatments. Furthermore, a band at 842 cm^{-1} was detected and tentatively correlated with either the V_3O_3 or the V_4O_2 structure in neutron-irradiated silicon.

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