

Effect of germanium doping on the formation kinetics of vacancy-dioxygen complexes in high dose neutron irradiated crystalline silicon

Peng Dong,^{1,2} Xuegong Yu,^{2,a)} Lin Chen,² Xiangyang Ma,² and Deren Yang^{2,a)}

¹Microsystem and Terahertz Research Center and Institute of Electronic Engineering, China Academy of Engineering Physics, Mianyang, 621999, People's Republic of China

²State Key Laboratory of Si Materials and School of Materials Science and Engineering, Zhejiang University, Hangzhou 310027, People's Republic of China

(Received 19 April 2017; accepted 21 August 2017; published online 5 September 2017)

The effect of germanium (Ge) doping on the formation kinetics of vacancy-dioxygen (VO₂) complexes in high dose neutron irradiated crystalline silicon (c-Si) has been quantitatively investigated using infrared spectroscopy at 10 K. It is observed that Ge doping of 10¹⁹ cm⁻³ enhances the formation of vacancy-oxygen (VO) complexes by ~15% during neutron irradiation and slightly suppresses the conversion of VO into VO₂ complexes. By studying the generation kinetics of VO₂ complexes in the temperature range of 300–345 °C, it is found that the activation energies of VO₂ generation are determined to be 1.52 and 1.71 eV in the reference and Ge-doped c-Si, respectively. According to the theory for diffusion limited reactions, it is suggested that Ge doping can retard the VO diffusion in c-Si and therefore reduce the capture probability of O_i for VO complexes. This may be attributed to the temporary trapping of vacancies by Ge atoms. Hence, the formation of VO₂ complexes in c-Si is slightly suppressed by Ge doping. *Published by AIP Publishing.*
[\[http://dx.doi.org/10.1063/1.5001139\]](http://dx.doi.org/10.1063/1.5001139)

I. INTRODUCTION

Germanium (Ge) doped crystalline silicon (c-Si) is of potential interest for applications in microelectronics and photovoltaic industries.^{1–3} Being electrically inactive in c-Si, the presence of Ge impurity does not affect its electrical properties. Ge atoms give rise to compressive elastic strains in the Si lattice, which can affect the reactions among vacancies, self-interstitials, and impurities. Thus, it could affect the formation of secondary defects in the course of irradiation and annealing. It has been found that an appropriate Ge-doping suppresses the formation of oxygen-related thermal donors and large-sized crystal originated particles (COPs).^{4,5} It also enhances the mechanical properties of c-Si wafers and mitigates the light-induced degradation of minority carrier lifetime of c-Si used for solar cells as well as enhances the oxygen precipitation, therefore improving the internal gettering capability of metallic contamination for devices.^{6–9}

Recently, Londos and Chreneos *et al.* have performed an extensive study of the influence of Ge doping on the behavior of oxygen and carbon impurity related complexes in electron irradiated c-Si, showing the potential application of Ge doped c-Si substrates in radiation-hardened microelectronics.^{2,10–15} They found that an appropriate Ge doping in c-Si crystals can effectively reduce the introduction rate of vacancy-oxygen (VO) complexes, which are generated by the pairing of oxygen atoms with mobile vacancies induced by irradiation.^{10,16,17} The VO complexes produce an acceptor level at $E_C - 0.17$ eV in the energy gap of Si and give rise to a well-known infrared (IR) band at 830 cm⁻¹.^{18,19} Acting as an important recombination center in Si, the VO complex

plays a critical role in the lifetime control of relative electronic devices. Upon thermal annealing above 300 °C, VO complexes can be transformed into VO₂ complexes by combining with interstitial oxygen (O_i). This gives rise to an IR band at 888 cm⁻¹ at room temperature. Although the VO₂ complex is electrically inactive, it may act as a nucleation center for oxygen aggregation processes. Therefore, VO₂ complexes are important for the development of oxygen precipitates.^{20,21} Londos *et al.* found that the conversion ratio of VO into VO₂ complexes decreased with the increase of Ge concentration in electron irradiated c-Si. This phenomenon is attributed to the elastic strains in the lattice induced by the Ge presence, which can affect the balance between two reactions, i.e., $VO + O_i \rightarrow VO_2$, $VO + Si_i \rightarrow O_i$.^{12,13,22} However, to the best of our knowledge, a detailed quantitative study on the effect of Ge-doping on the formation kinetics of VO₂ complexes has been little undertaken, including its generation rate and activation energy. Therefore, this study is very important for the physical picture of $VO \rightarrow VO_2$ transformation in Ge-doped c-Si.

In this paper, new light is shed on the role of Ge impurity atoms on the formation kinetics of VO₂ complexes in neutron irradiated c-Si with the help of Fourier transformed infrared absorption spectroscopy (FTIR). Compared with electrons, neutron irradiation mainly causes displacement damage in silicon bulk and therefore avoids the influence of ionization damage on the formation of point defects arising from displacement damage. It is found that Ge doping can lead to the increase of energy barrier for VO diffusion and thus slow down the formation of VO₂ complexes. These results give the deep understanding of the reaction among vacancy, interstitial oxygen, and Ge impurity atoms, which is necessary for the defect engineering of c-Si.

^{a)}Electronic addresses: yuxuegong@zju.edu.cn and mseyang@zju.edu.cn

II. EXPERIMENTAL DETAILS

In this study, two gallium-doped p-type Czochralski-grown Si crystals having 200 mm-diameter and $\langle 100 \rangle$ -orientation were investigated. One of the Si crystals was co-doped with $\sim 10^{19} \text{ cm}^{-3}$ Ge (GCz) and the other one was free from Ge (Cz). The both crystals were grown under the same pulling conditions, thus having similar thermal histories, resistivity, and interstitial oxygen contents. The concentrations of gallium and oxygen in both substrates were estimated to be 1.0×10^{16} and $10.4 \times 10^{17} \text{ cm}^{-3}$, respectively. The carbon concentration was below the detection limit of infrared spectrometer ($1 \times 10^{16} \text{ cm}^{-3}$). It is to mention that the gallium concentration was derived from the resistivity measurement by the four probe method. The oxygen (1106 cm^{-1}) and carbon (604 cm^{-1}) concentrations were determined by an FTIR spectrometer (IFS 66 V/S) at room temperature with the calibration coefficients of 3.14×10^{17} and $1.0 \times 10^{17} \text{ cm}^{-2}$, respectively. All the samples were mechanically polished with a thickness of $\sim 2 \text{ mm}$. Thereafter, they were subjected to neutron irradiation with a dose of $5 \times 10^{17} \text{ cm}^{-2}$ at room temperature. Subsequently, the samples were annealed from 300 to 345°C with a step of 15°C . Afterwards, the samples were dipped in the diluted HF solution to remove the surface oxide films. Finally, the VO and VO_2 complexes in all annealed samples were investigated using the FTIR spectroscopy by monitoring the relevant 836 and 896 cm^{-1} bands of IR spectra at 10 K. Herein, the absorption coefficient of VO complexes was determined by integrating the IR peak after separating the overlapped peaks by means of Lorentzian profile fitting.

III. RESULTS AND DISCUSSION

Figure 1 shows segments of the absorption spectra at 10 K for the neutron irradiated samples before and after isochronal anneals from 250 to 400°C , in steps of $\Delta T = 50^\circ\text{C}$ and $\Delta t = 30 \text{ min}$. The depicted bands at 836, 896, 924, 830, and 843 cm^{-1} have already been reported and assigned to VO, VO_2 , $[\text{VO} + \text{O}_i]$, V_2O , and V_3O complexes, respectively.^{23–25} All the IR bands at 10 K originating from these defects show a slightly blue shift of $6\text{--}8 \text{ cm}^{-1}$ as compared to those at room temperature. The concentrations of O_i before and after irradiation and the respective VO production in Cz and GCz silicon are illustrated in Table I. Upon irradiation, a large amount of the vacancies that survive annihilation with self-interstitials are captured by oxygen atoms to form VO pairs, since interstitial oxygen moves much slower than vacancy and therefore can be regarded as practically immobile compared with vacancy.^{26,27} This in turn results in the decrease of O_i concentration depicted in Table I, which is in agreement with the previous report.¹⁶ With the calibration coefficient of $6.25 \times 10^{16} \text{ cm}^{-2}$, the concentrations of VO complexes in Cz and GCz Si are determined to be 4.7×10^{16} and $5.4 \times 10^{16} \text{ cm}^{-3}$, respectively. The Ge-doping with a concentration of $\sim 10^{19} \text{ cm}^{-3}$ weakly enhances the VO production during high dose neutron irradiation, which is consistent with the previous reports by Londos and Chronos *et al.*^{10,16,17} The enhanced VO production can be ascribed to the Ge atoms which act as the temporary traps for vacancies and thus reduce the recombination rate of Frenkel

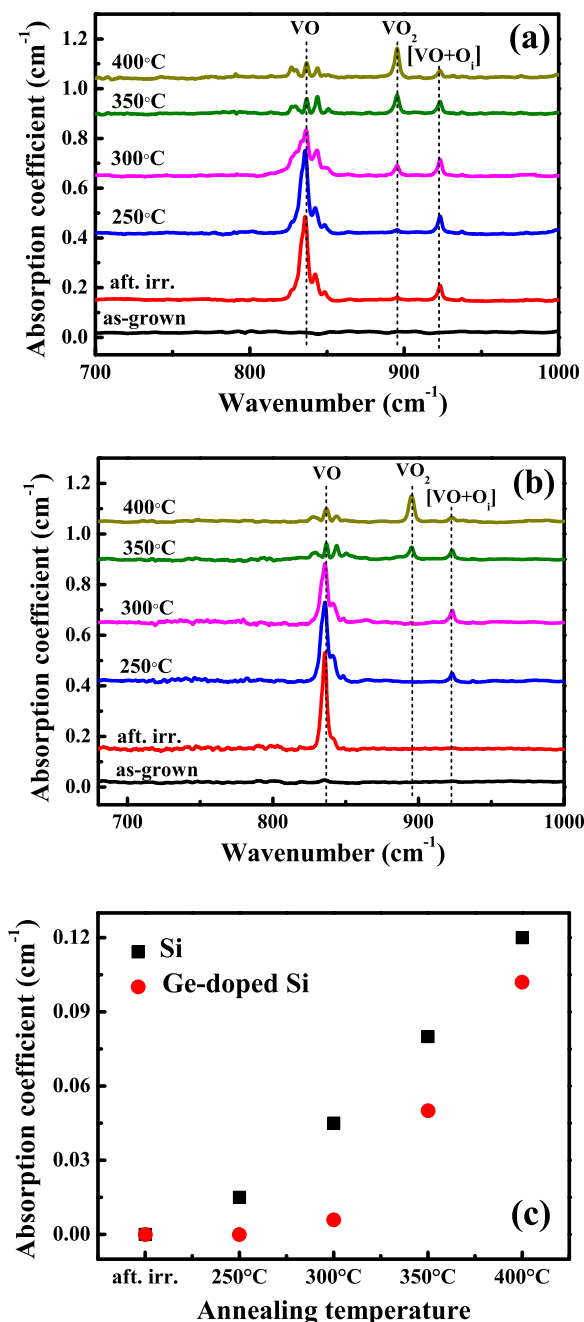


FIG. 1. Segments of the absorption spectra at 10 K for the irradiated Cz (a) and GCz Si (b) before and after isochronal anneals from 250 to 400°C , in steps of $\Delta T = 50^\circ\text{C}$ and $\Delta t = 30 \text{ min}$. (c) The production of VO_2 complexes in Cz and GCz Si.

pairs. With an increase of annealing temperature, the VO_2 related band at 896 cm^{-1} obviously grows at temperatures above 300°C [as depicted in Fig. 1(c)], whereas the absorption coefficient of 836 cm^{-1} band from VO complexes decreases

TABLE I. The concentrations of O_i before and after irradiation ($[\text{O}_i]_0$ and $[\text{O}_i]_{\text{a.i.}}$) and VO production ($[\text{VO}]$) in Cz and GCz silicon.

	[Ge] $\times 10^{19} \text{ cm}^{-3}$	$[\text{O}_i]_0$ $\times 10^{17} \text{ cm}^{-3}$	$[\text{O}_i]_{\text{a.i.}}$ $\times 10^{17} \text{ cm}^{-3}$	$[\text{VO}]$ $\times 10^{16} \text{ cm}^{-3}$
Cz Si	0	10.4	9.5	4.7
GCz Si	1.0	10.4	9.3	5.4

simultaneously. Therefore, it is inferred that the VO_2 formation can be ascribed to the capture of mobile VO complexes by O_i . It can be seen from Fig. 1(c) that VO_2 production is slightly suppressed in GCz Si than Cz Si during the isochronal anneals from 250 to 400 °C. Besides, the 924 cm^{-1} band from $[\text{VO} + \text{O}_i]$ complexes appears in the IR spectrum of Cz Si,^{25,29} whereas is absent in GCz Si with no annealing. This might be attributed to the suppression of Ge doping on the reaction between VO and O_i during irradiation at room temperature. The 924 cm^{-1} band from $[\text{VO} + \text{O}_i]$ complexes becomes more prominent after the subsequent annealing at 250 °C, but it begins to anneal out at a temperature above 300 °C, along with the appearance of 896 cm^{-1} band from stable VO_2 complexes. This suggests that the $[\text{VO} + \text{O}_i]$ complex is an intermediate structure for the conversion of VO into the VO_2 complex.^{25,29} Additionally, the IR bands at 830 cm^{-1} and 843 cm^{-1} from V_2O and V_3O complexes, respectively, are gradually annealed out in the temperature range of 350–400 °C. This might be associated with the dissociation of V_2O and V_3O complexes into vacancies and O_i .

Figure 2 shows the concentrations of VO_2 complexes as a function of annealing time in the temperature range of 300–345 °C. These are deduced from the peak amplitudes of the associated IR band at 896 cm^{-1} with the calibration coefficient of $4.25 \times 10^{16}\text{ cm}^{-2}$.³⁰ The concentration of VO_2 complexes

increases quickly at the early stage of annealing and then gradually reaches a saturation value after several hours. It can be seen that VO_2 complexes are relatively more difficult to generate in GCz Si than Cz Si. On the other hand, the concentration of VO complexes decreases with annealing time and accompanied by the growth of O_i concentration (as depicted in Fig. 3). After the complete annealing sequence, the decrease and increase of VO and O_i concentration, respectively, are slightly higher in GCz Si than Cz Si. The physical process behind this phenomenon will be discussed later.

The concentration of VO_2 complexes exhibits an exponential increase with time, and its formation rate can be expressed as

$$[\text{VO}_2](t, T) = [\text{VO}_2]_{\text{sat.}} [1 - \exp(-R(T)t)], \quad (1)$$

where T is the absolute temperature, t is the annealing time, $[\text{VO}_2]$ is the concentration of VO_2 complexes, and $R(T)$ is a temperature dependent rate constant. This relation holds for all annealing temperatures in both Cz and GCz Si.

The conversion ratios of VO into VO_2 complexes in Cz and GCz Si at different temperatures are depicted in Fig. 4, where $[\text{VO}]_{t=0}$ is the concentration of VO complexes in as-irradiated Si, and $[\text{VO}_2]_{\text{sat.}}$ is the saturation value for VO_2 concentration and derived from Fig. 2 at the temperature

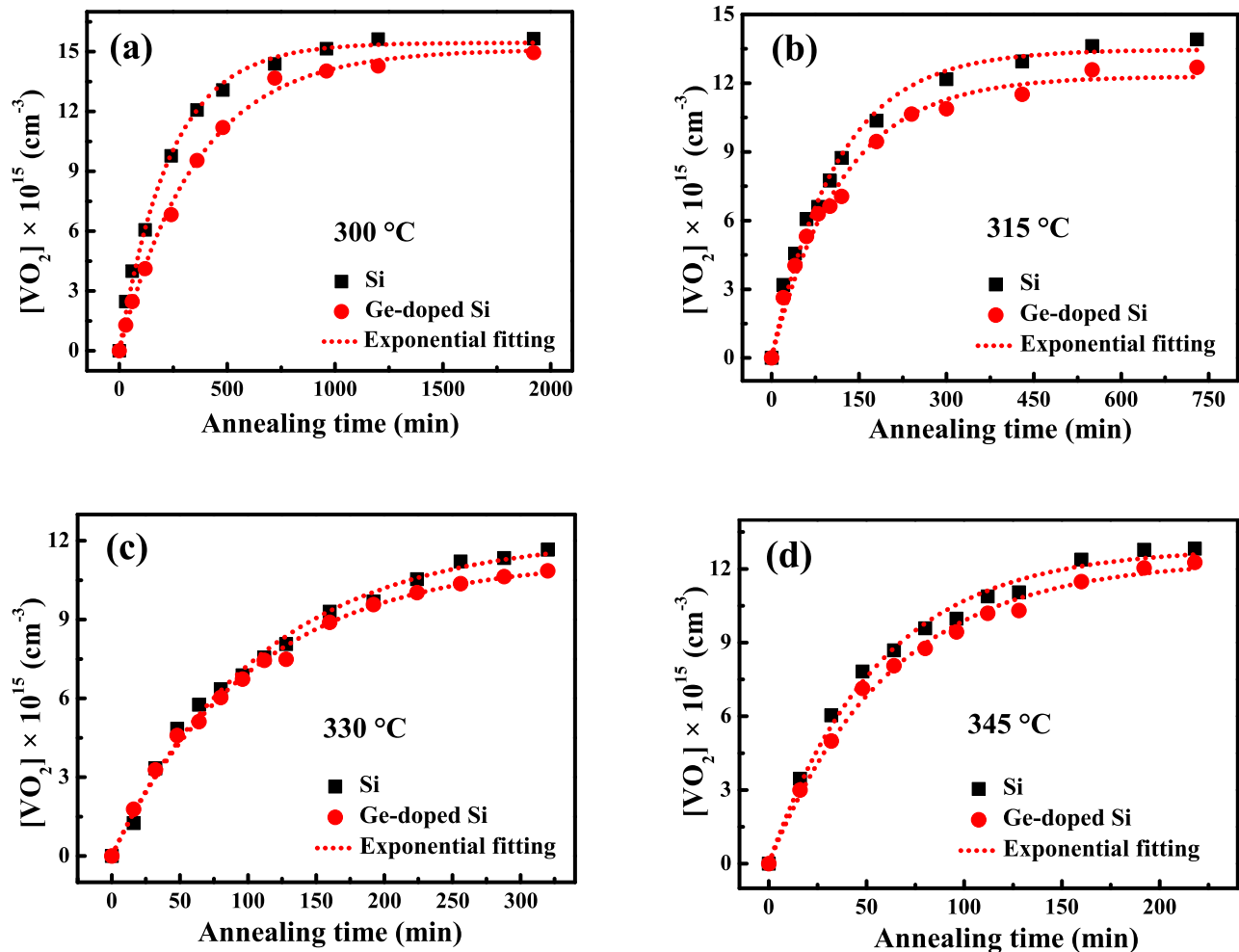


FIG. 2. The concentration of VO_2 complexes during isothermal annealing at (a) 300, (b) 315, (c) 330, and (d) 345 °C in irradiated Cz and GCz Si.

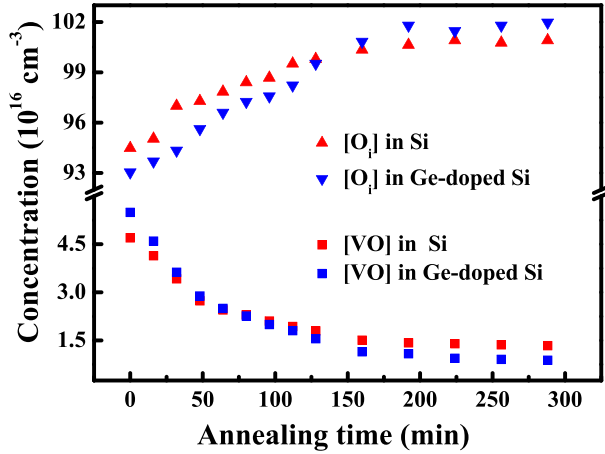


FIG. 3. The concentrations of O_i and VO complexes as a function of post-annealing times at 330 °C in Cz and GCz Si.

range of 300–345 °C. The $[VO_2]_{\text{sat}}$ values at 370 °C are derived from the IR spectra of Cz and GCz silicon annealed at 370 °C for 1 h, which is considered as the saturation state for VO_2 generation. It can be seen that about 20%–35% of VO complexes are transformed into VO_2 complexes. At the same time, the other two reactions are supposed to be involved in VO elimination, i.e., $VO + Si_i \rightarrow O_i$, $VO \rightarrow V + O_i$. Moreover, at all annealing temperatures, the conversion ratios of $[VO]_{t=0}$ to $[VO_2]_{t \rightarrow \infty}$ are lower in GCz Si as compared to Cz Si. Londos *et al.* have also found the similar changing trend.^{12,13} Apparently, the conversion reaction $VO + O_i \rightarrow VO_2$ is directly proportional to the oxygen concentration in Si. The higher the oxygen concentration, the stronger will be the conversion of VO into VO_2 complexes. However, there is no essential difference of oxygen concentrations between Cz and GCz Si. Therefore, it is suggested that Ge doping should be responsible for the suppressed transformation of VO into VO_2 complexes in c-Si. Generally, the interstitial silicon atoms (Si_i) induced by high dose of neutron irradiation tend to form cluster in heavily irradiated Cz Si. The compressive strain in crystalline lattice induced by Ge doping probably suppress the clustering of Si_i . Therefore, it is suggested that more free Si_i existing in GCz Si promotes the reaction $VO + Si_i \rightarrow O_i$, which in turn

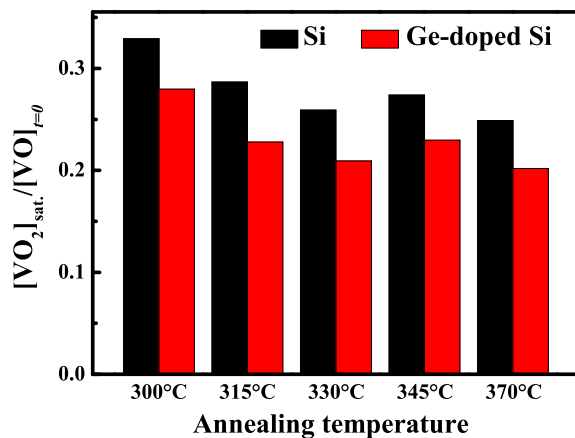


FIG. 4. The conversion ratios of VO to VO_2 complexes as a function of annealing temperatures in Cz and GCz Si.

suppresses the conversion reaction $VO + O_i \rightarrow VO_2$. This can be indirectly verified by the slightly enhanced VO loss and O_i generation after the annealing sequences in Ge-doped silicon (as illustrated in Fig. 3).

Figure 5 depicts the generation rate constant of VO_2 complexes as a function of temperatures in Cz and GCz Si. The formation rates of VO_2 complexes are relatively smaller in GCz Si as compared to Cz Si, which indicates that Ge doping slows the VO_2 formation in c-Si. For a singly activated process, an Arrhenius behavior is expected,

$$R_{VO_2} = \kappa_0 \exp\left(-\frac{\Delta E}{k_B T}\right), \quad (2)$$

where k_B is the Boltzmann's constant, ΔE is the activation energy, and κ_0 is the pre-exponential factor. From the slope of $\ln R \sim 1/T$, the activation energies for the VO_2 formation are determined to be 1.52 and 1.71 eV in Cz and GCz Si, respectively. The formation of VO_2 complexes can be expressed by the reaction $VO + O_i \rightarrow VO_2$. According to the theory for diffusion limited reactions,³¹ the reaction rate is proportional to $D_{VO} + D_{O_i}$, where D_{VO} and D_{O_i} represent the diffusivity of VO and O_i , respectively. However, according to the reported diffusion coefficients for interstitial oxygen and VO complexes,^{27,32} the diffusion rate of O_i at 330 °C is estimated to be $\sim 1.0 \times 10^{-22}$ cm²/s and is far below that of VO complexes (5.6×10^{-15} cm²/s). Hence, interstitial oxygen can be regarded as practically immobile at temperatures of 300–345 °C. Therefore, the formation of VO_2 complexes can be ascribed to a process of VO complexes diffusing towards O_i . And the diffusion energy barriers of VO complexes are 1.52 and 1.71 eV in Cz and GCz Si, respectively. These are close to the previously reported values of 1.7–1.8 eV extracted from FTIR and deep-level transient spectroscopy (DLTS) studies.^{32,33} It is suggested that a larger energy barrier in Ge-doped Si leads to the decrease of VO diffusivity, which in turn slows down the capture of VO by O_i and therefore retards the VO_2 formation in c-Si.

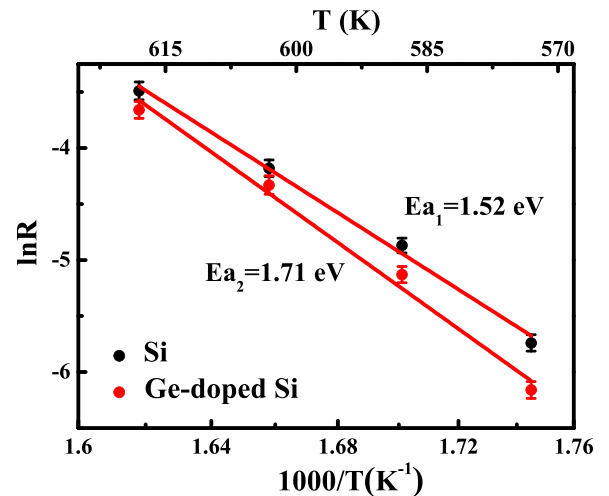


FIG. 5. The generation rates of VO_2 complexes as a function of temperatures in neutron irradiated Cz and GCz Si. The solid lines are obtained by a linear fit of experimental points.

It is proposed by Furuhashi and Taniguchi that vacancy migration is involved in the diffusion of VO complexes.³⁴ They have suggested that the VO complex first transforms into a bond-center structure, in which a vacancy is the nearest neighbor to Si–O–Si. Thereafter, the vacancy migrates on each Si site step by step along a six-member ring and becomes the nearest neighbor to the Si–O–Si structure again. Finally, the bond-center structure newly forms VO after the vacancy jumps into the Si site nearest to the oxygen, and therefore completes an atomic step of VO diffusion.³⁴ It is speculated that the suppressed VO diffusion in GCz Si might be related to the vacancy migration along a six-member ring. The energy diagram for VO diffusion in GCz silicon is shown in Fig. 6. Hybrid density functional theory calculations show that the binding energy of GeVO complexes is 0.21–0.23 eV larger than the counterpart of VO complexes, indicating that the system gains more energy by the formation of GeVO complexes as compared to VO complexes.³⁵ These GeVO complexes have been found by deep level transient spectroscopy in electron irradiated Cz-grown Si_{1-x}Ge_x crystals. However, in the present study, the Ge-doping concentration is not high enough to cause a visible shift of the 836 cm⁻¹ band in IR spectra. In view of the above-mentioned analysis, it is inferred that VO complexes are more inclined to form at the neighboring site of Ge atoms. The vacancy migration involved in VO diffusion may be affected by its neighboring Ge atom. During vacancy migration, the neighboring Ge atom serves as a temporary sink for a vacancy and gives rise to the metastable GeV pair with the binding energy of 0.25–0.27 eV.^{35,36} The captured vacancy has to overcome the additional binding energy to escape from the neighboring Ge atom (from A to B in Fig. 6). Coincidentally, the reported binding energy of GeV pair (0.25–0.27 eV) is close to the energy barrier difference of VO diffusion (0.19 eV) between Cz and GCz Si. Therefore, the retarded VO diffusion in Ge-doped Si is tentatively ascribed to the temporary trapping of a vacancy by the Ge atom associated with the formation of GeV pair. However, the physical picture for the VO diffusion in Ge-doped Si is complicated and further investigations are required to clarify the issue.

IV. CONCLUSIONS

FTIR measurements have been performed to quantitatively study the formation kinetics and mechanism of VO₂

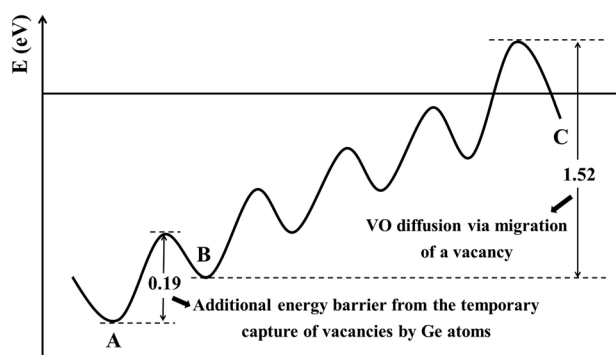


FIG. 6. The schematic illustration for the suppressed VO diffusion in GCz silicon.

complexes in neutron irradiated Ge-doped Si. It is found that Ge-doping with a concentration of $\sim 10^{19}/\text{cm}^3$ slightly enhances the VO formation in c-Si and suppresses the transformation of VO into VO₂ complexes, which might originate from the high concentration of self-interstitial Si atoms in GCz Si, thus promoting the reaction of $\text{VO} + \text{Si}_i \rightarrow \text{O}_i$. Besides, the activation energies for VO₂ formation are determined to be 1.52 and 1.71 eV in Cz and GCz Si, respectively, which corresponds to the energy barriers of VO diffusion according to the theory for diffusion limited reactions. The larger energy barrier of VO diffusion in GCz Si suppresses the diffusion of VO complexes, thus, slows down the capture of VO complexes by O_i and retards the VO₂ formation. Furthermore, it is found that the increase in energy barrier of VO diffusion by Ge-doping coincides with the binding energy of GeV pairs. This led us to assume that the retarding effect of VO diffusion in GCz Si might be due to the temporary trapping of a vacancy by its neighboring Ge atom, that is, the formation of GeV pair.

ACKNOWLEDGMENTS

The authors would like to thank the Science Challenge Project (No. TZ2016003-1) and the National Natural Science Foundation of China (Nos. 61604139, 51532007 and 61574124) for financial support.

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