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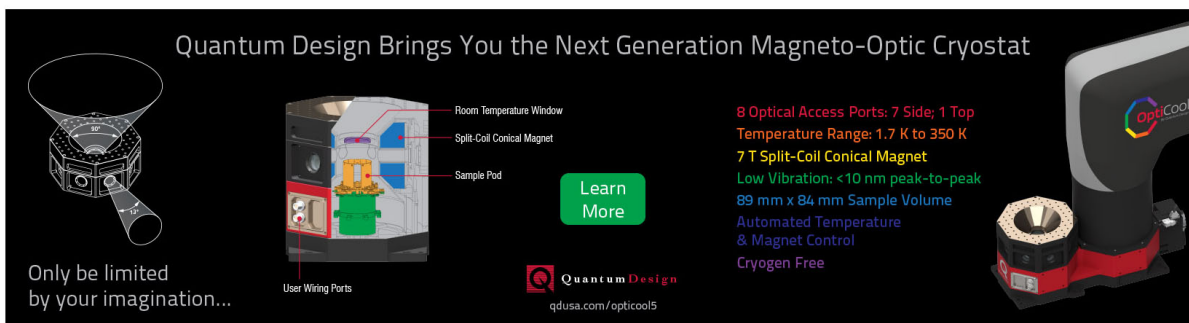
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On the manifestation of phosphorus-vacancy complexes in epitaxial Si:P films

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In situ doped epitaxial Si:P films with P concentrations $>1 \times 10^{21}$ at./cm³ are suitable for source-drain stressors of n-FinFETs. These films combine the advantages of high conductivity derived from the high P doping with the creation of tensile strain in the Si channel. It has been suggested that the tensile strain developed in the Si:P films is due to the presence of local Si₃P₄ clusters, which however do not contribute to the electrical conductivity. During laser annealing, the Si₃P₄ clusters are expected to disperse resulting in an increased conductivity while the strain reduces slightly. However, the existence of Si₃P₄ is not proven. Based on first-principles simulations, we demonstrate that the formation of vacancy centered Si₃P₄ clusters, in the form of four P atoms bonded to a Si vacancy, is thermodynamically favorable at such high P concentrations. We suggest that during post epi-growth annealing, a fraction of the P atoms from these clusters are activated, while the remaining part goes into interstitial sites, thereby reducing strain. We corroborate our conjecture experimentally using positron annihilation spectroscopy, electron spin resonance, and Rutherford backscattering ion channeling studies. © 2016 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4942605>]

The use of source-drain (S/D) stressors to boost the carrier mobility is a well-known scaling approach to improve the transistor performance. SiGe S/D stressors are preferred for p-FinFETs,¹ whereas Si:C(P) or Si:P S/D stressors are preferred for n-FinFETs.^{2,3} The use of Si:P S/D stressors is a more recent approach wherein the high amounts of P ($>1 \times 10^{21}$ at./cm³) enable to combine the advantages of a high conductivity with strain introduction in the channel.⁴ Even though recent works describe the obtained performance improvement for n-FinFETs, not many reports are available on the fundamental understanding of the properties of the Si:P films in relation to the electrical characteristics. The mechanism of the conductivity enhancement upon annealing still needs to be explained. Further, it is unclear whether the tensile stress observed in the Si:P films is due to pure differences in the covalent radii between P and Si or due to the formation of specific local phases/complexes.

Ye *et al.*⁵ suggested that the strain observed in the epitaxial Si:P films might be due to local clustering of P into stable pseudocubic Si₃P₄ clusters, which do not contribute to the conductivity. After the exposition to a submelt millisecond laser annealing, these local clusters would be dispersed resulting in an increase in the conductivity. Similarly, it was shown by Rosseel *et al.*⁶ that upon annealing the resistivity decreased by 50% versus the one for the as grown Si:P films due to an increase in the activated dopants. For example, in Si:P with 4.6% total P, micro-Hall measurements indicated an increase

in carrier concentration from an initial 1.58×10^{20} at./cm³ to 9.8×10^{20} at./cm³ in the annealed layer.⁶ Moreover, both groups reported a small (<1%) decrease in lattice strain relative to the as grown layer, after post epi anneal as measured by High Resolution X-Ray Diffraction (HR-XRD). The strain reduction increased with thermal budget of the anneal and was attributed to the dispersion of the Si₃P₄ clusters.^{5,6} However, none of these publications proved/verified the existence of the Si₃P₄ phase neither experimentally nor theoretically. In this work, we examine the nature of the P configuration in Si:P films using *ab-initio* simulations and investigate the possibility of forming local Si₃P₄ clusters in the epitaxial layers. We corroborate the results of the *ab-initio* simulations with Positron annihilation studies (PAS), Electron Spin Resonance (ESR), and Rutherford Backscattering Spectroscopy (RBS) ion channeling.

In this paper, we try to answer if the reported conductivity and strain behaviors for the Si:P films are due to the formation/dissolution of Si₃P₄ clusters. The pseudocubic Si₃P₄ structure is a defective zinc blende.⁷ From a vacancy centered perspective, four P atoms bound to the vacancy are present as second nearest neighbors. To avoid any confusion, we refer within the body of this article to the vacancy centered Si₃P₄ as a P₄V complex.⁸ We investigate the probability of its formation using *ab-initio* studies by evaluating the possibility that the P atoms can exist as close nearest neighbor atoms (such as 2nn). Based on these calculations, we also derive the probability for the formation of the Phosphorus–Vacancy (P–V) complexes. Our results show that it is not energetically favorable that the P atoms cluster by themselves into any nearest neighbor arrangement due to their coulombic repulsion. However, the presence of vacancies can induce the P atoms to cluster

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around these, giving rise to an E center (dopant bound to a vacancy defect forming a deep level in the bandgap). These vacancy centers disappear after annealing concurrent with an improvement in the conductivity. With the temperature/time budget of our anneals (1200 °C/0.5 ms with 2 pulses), not all of the P atoms complexed with vacancies are substitutionally dissolved, as demonstrated by the ESR measurements. Moreover, it appears that, based on the increased channeling yield from RBS measurements, a fraction of the undissolved P atoms goes into interstitial positions. This displacement of P atoms to interstitial sites also explains the loss of strain for the annealed layers as measured by HR-XRD.⁶

Details on the epitaxial growth of Si:P are described in Ref. 6. For the growth conditions used, a maximum P concentration of 4.6% can be obtained, while retaining a fully strained epitaxial film. For concentrations beyond 4.6%, the roughness of the epitaxial layers increases and the film undergoes an epitaxial breakdown. Hence, we restrict our study on the local clustering phenomena for P to a maximum P concentration of 4.6%. The *ab-initio* simulations were carried out using the quantum espresso⁹ package combined with Vanderbilt pseudopotentials. A 340 eV kinetic energy cut off and gamma k point mesh sampling were used, combined with Perdew–Burke–Ernzerhof (PBE)¹⁰ exchange correlational function. The model used consists of a 216 atoms silicon unit cell in which two P atoms (labelled as P1 and P2) were randomly placed at different radial distances from each other. The atomic positions of the structure were then relaxed, and the resulting total energy was calculated for the different configurations. The change in total energy with respect to the reference energy, corresponding to different configurations is extracted and shown in Fig. 1(a).

As evident from Fig. 1(a), the concomitant presence of two P atoms in their immediate vicinity leads to an increase in the total energy. When the P atoms occupy positions farther and farther apart, the total energy of the system decreases. The kink computed for a radial distance at 5.9 Å is attributed to a specific configuration where two P atoms lie in parallel planes that leads to a maximum stress transfer.¹¹ As the system considered is confined to 216 atoms, the maximum

distance to which the second P atom (P2) can be positioned from the first one (P1) without interfering with the imposed periodic boundary conditions, is 12 Å, and the total energy of the system at 12 Å is defined as the reference minimum energy. Whenever the difference in total energy between the considered configurations and the reference one (P2 located at 12 Å from P1) is lower or equal to the thermal energy at 300 K, i.e., 0.026 eV, we determine the configuration to be stable. It is found that, when P atoms are located at least 6.6 Å from each other, the difference in energy becomes close to the thermal energy, suggesting that this configuration is possible at room temperature. This suggests that energetic considerations favor the distributions with P atoms separated at least 4 atomic sites (or more) from each other. Similar trends have been found when performing the simulations for different P doping concentrations ($\leq 4.6\%$). The P atoms always favor to stay as far away as possible from each other. In view of their similarity, we restrict the remaining discussion to layers with the 4.6% P doping concentration.

It must be noted that unlike Si:C,¹² which has a distinct energy minimum for a 3nn distribution of C atoms,¹¹ there is no such minimum energy position for the distribution of P atoms. Although P atoms give rise to tensile stress, when they are present in an ionized form, the coulombic repulsion prevents any sort of nearest neighbor arrangement. Thus, in the as grown samples and in the absence of defects/vacancies, P does not like to have another P atom as a close nearest neighbor.

The situation becomes different if a vacancy is inserted in the system. Indeed for the combination of a single P atom with a vacancy, the total energy of the system decreases when the distance between the P atom and the vacancy is reduced. This affinity of P towards vacancies is similar to the one noted previously by Ganchenkova *et al.*¹³ The formation enthalpy of a P_nV complex with $n = 1$ is positive and equals to 3.4 eV, and it decreases as the number of P atoms (n) increases. Consequently, the formation enthalpy of the P_4V complex, where the vacancy is surrounded on all sides by a P atom, is found to be negative (−0.31 eV), indicating that its formation is a spontaneous process from a thermodynamic perspective. (A similar behavior for the formation of P_4V complex is also reported by Chen *et al.*¹⁴ in their studies on gettering.) The question arises whether two such P_nV clusters can co-exist next to each other forming a divacancy configuration. The enthalpy of formation for two such configurations, where the two P_3V clusters are located as 3nn (Fig. 1(b)), has indeed negative energetic values (~ -0.2 eV and ~ -0.6 eV) indicating their stability. Hence, our finding suggests that, from an energetic perspective, P_nV clusters could be present as both monovacancies and divacancies in the Si:P lattice.

The density functional theory (DFT) results obtained on the P distribution and P-V complexes distribution within the silicon matrix are corroborated by PAS measurements to understand the nature of the vacancies that are formed in the Si:P layers. For a detailed description on Positron annihilation spectroscopy, the reader is referred to Refs. 15 and 16. We used Doppler broadening (DOBS) and Coincidence Doppler Broadening Spectroscopy (CDOBS) to study the vacancy defects in the Si:P-layers. For DOBS, we used a

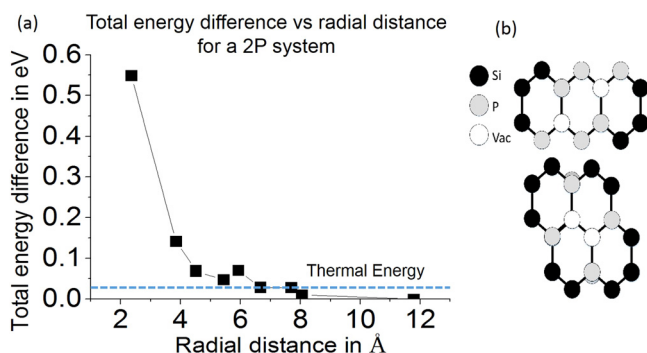


FIG. 1. (a) Evolution of the total energy difference for substitutional P atoms in the Si lattice with respect to different radial distances in the 2P system. As from a minimum separation distance of 6.6 Å, all the values are below the thermal energy. (b) The two possible configurations of divacancies (for 4.6% doping) seen in the (110) direction. The configuration shown on the top has an enthalpy of formation equal to −0.16 eV, whereas the one below has an enthalpy of −0.6 eV.

variable positron implantation energy for depth profiling. In order to improve the sensitivity for the layer, the surface oxide was removed by etching the samples in HF acid prior to the measurements.^{17,18}

Fig. 2 shows the CDOBS ratio curves for the 4.6% P sample (as-grown, annealed) together with the results for an E-center (P-V) and a divacancy in silicon. An e^+ -trap-free silicon sample was used as a reference for normalization. The S and W parameters^{15,16} were first measured as a function of positron implantation energy (DOBS) and the corresponding W(S) plot for the 4.6% Si:P layer is shown in the inset. The arrows in the inset indicate the direction of increasing positron implantation energy (1, 2 indicating the lowest implantation energy). The circled points are characteristic for the dominating defects and correspond to the maximum S parameter observed in the layers. The e^+ -diffusion length in the as-grown layers is very short owing to P-V complexes formed by the high P concentration. A sharp turning point from the surface-Si:P-layer-line to the Si:P-layer-bulk line confirms this and therefore suggests that the e^+ -trapping is in saturation or close to saturation in the respective layers.

The CDOBS measurements were carried out at an implantation energy of 1.3 keV that yields a narrow implantation profile with a mean e^+ -implantation depth in the middle of the Si:P layer. The Si:P 4.6% samples fall in between the P-V and V_2 ,^{19,20} with the characteristic momentum peak at 1.2 a.u. being slightly higher in the annealed sample and closer to the P-V center. Previous studies²¹ indicate that adding more P atoms to a vacancy in Si decreases the intensity at high momenta and hence also the W parameter (inset in Fig. 2). Hence, this suggests that the dominating defect in the as-grown layers is most likely a monovacancy decorated with several P atoms. However, divacancy-sized complexes also

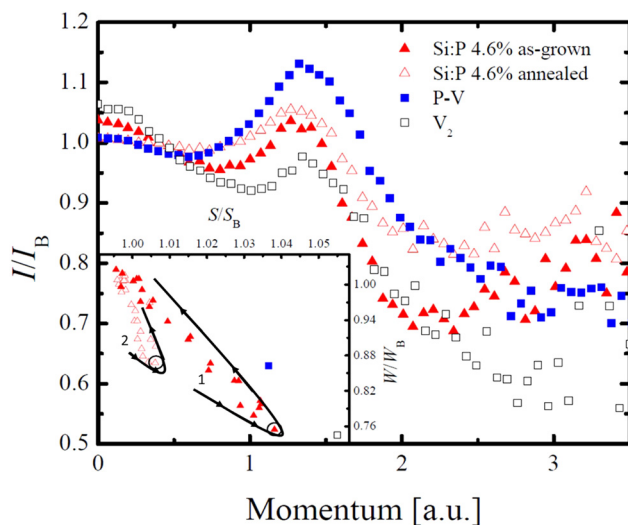


FIG. 2. CDOBS ratio curves for the 4.6% P sample (as grown, annealed) together with the results for an E-center (P-V)²¹ and a divacancy^{19,20} in silicon. The inset shows the W(S)-plot for the 4.6% P sample. The arrows in the inset represent the direction of increasing positron implantation energy starting with (1) for the as-grown layer and (2) for the annealed one. The two circles indicate the maximum S parameter obtained for the as grown and annealed layers. The dominant defects in the as grown layers are P-V complexes. In CDOBS curve, the increase in intensity at high momenta is a clear indication of the reduction in vacancies as a result of the laser anneal.

cannot be ruled out. Annealing reduces the concentration of the vacancy defects, indicated by a reduction in the S parameter of the layer and the increase in intensity at high momenta in the CDOBS spectrum.

The exact mechanism responsible for the reduction in vacancies, resulting from the post-epi annealing process is not understood. Here, we will restrict ourselves to the qualitative interpretation of the increase in conductivity and the loss in strain reported for the annealed Si:P films.

To assess the atomic nature of occurring point defects, the location (site properties) and the distribution of P atoms, K-band (~ 20.4 GHz) ESR has been carried out at 4.3 K. (for details, see Ref. 22). For the annealed Si:P film (Fig. 3), the only signal observed from the sample itself is a weak doublet, of splitting $a = 42.0 \pm 0.4$ G and centered at $g = 1.99866$. It is the well-known doublet stemming from unpaired electrons at P atoms that have taken the substitutional doping sites in c-Si. The doublet splitting arises from the hyperfine interaction of the localized unpaired electron wave function with the nuclear spin ($I = 1/2$) of the ^{31}P isotope (100% natural abundance) nuclei. The as-grown films were excessively lossy,²³ so no reliable ESR data could be attained. The doublet, of low intensity, indicates that a small portion of the P atoms has taken the dopant-active position as a result of the annealing. The reduced intensity of the doublet compared to the reference marker at $g = 2.00229$ clearly points out that the number of activated P atoms constitute a fraction (50%) of the total P atoms after anneal. The ESR finding is also in line with the observation that 50% of the dopant atoms are activated by laser anneal as noted by Rosseel *et al.*⁶ This poses a fundamental question regarding the location of the remaining fraction of the P atoms complexed to vacancies.

We propose that after annealing, the remaining of the P atoms attached to the mono and divacancies have reached the interstitial sites, leading to a reduction in the vacancy defects. This proposal is supported by the increase in the RBS channeling yield for the annealed Si:P films. The channeling yield for the as-grown 4.6% Si:P films is measured to be 7.9%, while it rises to 17.6% after laser annealing, which is almost a factor of 2 higher than for the as-grown films, suggesting a

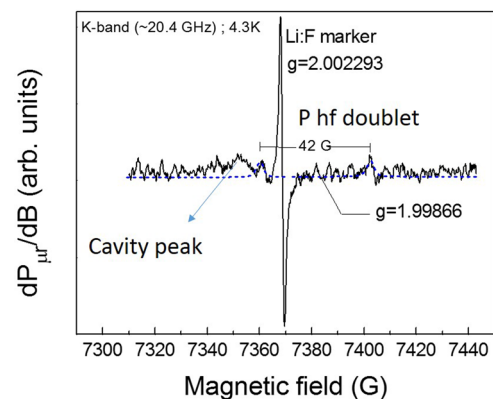


FIG. 3. K-band ESR spectrum of an annealed Si:P sample with 4.6% P doping. The narrow central signal at $g = 2.002293$ stems from an added Li:F marker sample, while the signal at the left of the doublet originates from cavity background. The reduced intensity of the P hyperfine doublet at $g = 1.99866$, compared with the reference marker, indicates that only a portion of the P atoms get activated after anneal.

substantial increase in P-interstitial concentration. The loss of strain after the post-epi treatments as described by Ye *et al.*⁵ and Rosseel *et al.*⁶ can be attributed to the loss of P atoms from lattice to interstitial sites and not necessarily to the dispersal of P₄V complexes. Both ESR as well as RBS suggests that most likely a fraction of the P atoms occupy interstitial sites.

As a summary, we have investigated the structure and lattice position of P atoms when incorporated in highly P-doped films used for S/D stressor applications in n-FinFET devices. The as-grown and annealed Si:P films were studied using first-principles calculations complemented with PAS, ESR, and RBS measurements. From the DFT studies, we inferred that the formation of isolated P₄V and P₃V clusters in a third nearest neighbor configuration is likely to occur, thanks to their negative enthalpies of formation. With PAS, we clearly observe the signatures of monovacancies, whereas the structure of divacancies with P decoration has precluded us from observing them, even if they had manifested. Upon laser annealing, the concentration of vacancies reduces. This reduction suggests that P_nV complexes can be broken up by the annealing process. As a result, a fraction of the P atoms complexed to P_nV moves to substitutional sites (as inferred from ESR measurements), whereas the remaining fraction of the P atoms either moves to interstitial sites or takes a relaxed atomic position from the substitutional sites, as evidenced from the channeling yield of RBS measurements and from the loss of strain measured by HR-XRD. Thus, the conductivity increases, and the reduction in strain is explained by the formation of interstitials from different P_nV complexes in the Si:P films.

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