Tailoring Superconducting Phases Observed in Hyperdoped Si:Ga for Cryogenic Circuit Applications

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Hyperdoping with gallium (Ga) has been established as a route to observe superconductivity in silicon (Si). The relatively large critical temperatures \(T_c\) and magnetic fields \(B_c\) make this phase attractive for cryogenic circuit applications, particularly for scalable hybrid superconductor–semiconductor platforms. However, the robustness of Si:Ga superconductivity at millikelvin temperatures is yet to be evaluated. Here, we report the presence of a reentrant resistive transition below \(T_c\) for Si:Ga whose strength strongly depends on the distribution of the Ga clusters that precipitate in the implanted Si after annealing. By monitoring the reentrant resistance over a wide parameter space of implantation energies and fluences, we determine conditions that significantly improve the coherent coupling of Ga clusters, therefore, eliminating the reentrant transition even at temperatures as low as 20 mK.

Superconducting silicon (Si) is considered a key to realizing of all-Si hybrid superconductor–semiconductor (S-Sm) devices that are ideal for highly scalable quantum circuits. Superconductivity in Si has been demonstrated by hyperdoping beyond metal–insulator transition limits via boron (Si:B) and gallium (Si:Ga). Moreover, hyperdoped Si:B has been successfully integrated into gate-tunable all-Si Josephson junctions (JJ's) and superconducting quantum interference devices (SQUIDs). Nevertheless, relatively low critical temperatures \(T_c < 800 \text{ mK}\) and magnetic fields \(B_c < 1 \text{ T}\) for superconducting phases of Si:B would possibly limit their application in quantum circuits, due to susceptibility to thermal and flux noise.

The superconducting phase in hyperdoped Si:Ga may be a plausible alternatives because of its larger \(T_c \sim 6 \text{ K}\) and \(B_c \sim 10 \text{ T}\). But Si:Ga transport properties at temperatures far below its \(T_c\) have not been explored. Such temperatures are commonly used for quantum devices in order to minimize the influence of thermal energy (\(~ k_B T\)) on the quantum two-level systems. Given that the superconductivity in Si:Ga has been attributed to the presence of a few nm-thick Ga layer segregated near the top surface, it is possible that a re-entrant resistive phase exists below \(T_c\) due to disorder and percolation induced by Si or SiO\(_2\) mixing. Therefore, it is critical to determine the superconducting characteristics for hyperdoped Si:Ga well below its critical temperature all the way down to mK range.

In this work, we study the influence of processing condition on the superconducting properties of hyperdoped Si:Ga prepared by Ga\(^{+}\) implantation. We first demonstrate that the implantation energy of 80 keV, which is used in all previous studies of Si:Ga superconductivity, leads to emergence of a re-entrant insulating state below \(T_c\). We then tailor the Ga distribution within the implanted region by tuning processing parameters such as implantation energy \(E_{\text{IMP}}\), dopant activation temperature \(T_{DA}\) and Ga\(_{\beta}\) fluence \(\Phi_{Ga}\). Although superconductivity is observed over a wide parameter space, resistive transition temperature \(T_c\) remains within a narrow range of \(5.2 - 7.1 \text{ K}\). This is in agreement with superconductivity stemming from confined crystalline (e.g., \(\beta\)-phase) or amorphous Ga with varying degrees of coherent coupling. Furthermore, we demonstrate conditions where the superconducting network in Si:Ga reaches the connectivity level necessary to maintain dissipationless conductivity at temperatures as low as 20 mK.

In order to prepare hyperdoped Si:Ga we started with undoped Si(100) wafers (\(\rho > 1000 \text{ \Omega cm}\)). A 30 nm thick SiO\(_2\) cap layer was deposited on each wafer using plasma-enhanced chemical vapor deposition (PECVD). Samples underwent Ga\(^{+}\) implantation at ambient temperature with energies \(E_{\text{IMP}} = 25 \text{ keV–80 keV}\), and fluences \(\Phi_{Ga} = 4 - 6 \times 10^{16} \text{ cm}^{-2}\). Implanted wafers then underwent dopant activation annealing at temperatures \(T_{DA}\) of 300 – 800 \(^\circ\text{C}\) for 1 min in \(N_2\) using a rapid thermal annealer.

Transport properties were initially evaluated by measuring differential resistance vs temperature down to 1.5 K in Van der Pauw (VdP) geometry (using an Oxford Instruments Teslatron PT measurement system). In Fig. 1a we show the superconducting characteristics of the Si:Ga chips implanted at \(E_{\text{IMP}} = 80 \text{ keV}\) and \(\Phi_{Ga} = 4 \times 10^{16} \text{ cm}^{-2}\); these parameters are adapted from previous reports of Si:Ga superconductivity in Ref. [6–8, 12]. Fig.1a shows the sheet resistance \(R_S\) normalized to its maximum value below 10 K \((R_{R_{\text{MAX}}})\), vs temperature for six superconducting samples with \(T_{DA}\) increasing from 575 to 700 \(^\circ\text{C}\). The resistive transition temperature \(T_c\) for the superconducting samples is shown in Fig.1b. \(T_c\) for each \(R_S(T)\) trace is defined as the temperature where \(R_S = 0.5 R_n\). As dopant activation temperature increases, \(T_c\) varies ± 0.8 K around the \(\beta\)-Ga \(T_c\). This is consistent with the solid-state precipitation of Ga within the implanted Si:Ga to form a superconducting Ga-rich network.
Figure 1. (a) Sheet resistance $R_S$ (normalized to its peak value below 10K, $R_{MAX}$) vs temperature for Si:Ga samples with implantation energy $E_{IMP}$ of 80 keV. (b) Resistive transition temperatures $T_c$ vs anneal temperature $T_{DA}$ for the samples shown in (a). (c) Superconducting transition of a Si:Ga sample with $E_{IMP}$ = 80 keV that is annealed at 650 °C. The inset shows transport behavior below 5 K (sub–$T_c$) marking the reentrant resistance ($R_r$) as a function of temperature $T_{DA}$.

In order to evaluate the superconducting behavior below $T_c$, we examined the temperature dependence of sheet resistance $R_S(T)$ in 1.5 K – 6 K temperature range. Fig 1 displays the $R_S(T)$ trace for the sample with $T_{DA} = 650$ °C. In the inset we show the emergence of a reentrant resistance as temperature approaches 1.5 K. In order to compare the extent of reentrant behavior between samples, we identified $R_S$ at 1.5 K as the characteristic reentrant resistance ($R_r$). In Fig 1 we show the dependence of $R_r$ on $T_{DA}$ for samples $E_{IMP}$ = 80 keV, where the lower $T_{DA}$ typically leads to smaller re-entrant resistance. The dramatic growth in $R_r$ above 625 °C may be attributed to the loss of Ga, leaving poorly-connected Ga networks behind.

For the sample with the largest reentrant resistance (i.e., $E_{IMP}$ = 80 keV and $T_{DA}$ = 700 °C), we investigated the structural characteristics using cross-sectional Transmission Electron Microscopy (TEM). Fig. 2(a) displays TEM images of the sample cross-section. Up to 100 nm below the SiO$_2$ cap, we observe nano-crystalline Si whose structure was recovered during the post-implantation annealing. Within this region, as well as at the Si/SiO$_2$ interface, a secondary phase is present in the form of ~5–10 nm wide clusters. These clusters appear to vary from crystalline to amorphous depending on the location within the implanted region. High-angle annular dark-field (HAADF) images and energy dispersive X-ray spectroscopy (EDS) elemental maps for the same cross-section, shown in Fig 2(b), confirm that the clusters are highly Ga-rich. Ga precipitation is highly likely in the Si:Ga system as Ga is only soluble in Si up to 0.1 at.% at 1000 °C. Particularly at elevated temperatures (e.g., 700 °C) insoluble Ga is expected to precipitate into clusters within the bulk or below the SiO$_2$ barrier. The more closely-packed clusters at Si/SiO$_2$ may form a pseudo-2D Ga thin film to host the superconductivity. However, the layer appears to be granular with regions bridges with few nm wide Si weak-links. Although those weak-links are highly p-doped, their carriers could eventually freeze in large fractions at $T < 1$ K. This will limit their ability to carry supercurrents through the proximity effect, as their superconductivity eventually vanishes at very low carrier concentration.

Therefore, the coherent coupling between superconducting Ga puddles can eventually be destroyed at near-zero temperatures.

To boost connectivity of Ga clusters, we lowered the Ga$^+$ implantation energy to less than 80 keV. In Fig 3(a), we show the Ga concentration vs depth calculated by the Transport of Ions in Matter (TRIM) software for $E_{IMP}$ = 25 – 80 keV at a fixed fluence of $\Phi_{Ga} = 4 \times 10^{16}$ cm$^{-2}$. By lowering the
implantation energy, Ga distribution peak becomes narrower and taller as it shifts toward the top surface. Accordingly, three sets of Si:Ga samples with EIMP = 25, 35, and 45 keV were prepared. At each implantation energy, samples underwent activation annealing at 350 to 800 °C. Fig. 3(b) displays the measured Tc, a function of TDA and EIMP, where lower energies on average lead to higher Tc,s, closer to values for α-Ga and γ-Ga. We note that superconductivity was absent in samples with EIMP = 25 keV, and for EIMP = 35 keV only annealing at 550 °C yielded superconductivity. This is consistent with the TRIM simulation results for EIMP < 45 keV; a larger portion of the Ga peak resides within the SiO2 cap (see Fig. 3(a)), therefore the amounts that remain in Si post-annealed are not sufficient to form a connected Ga network. Fig. 3(c) shows the reentrant resistance Rr for the superconducting samples vs TDA and EIMP. Increasing the annealing temperatures above 650 °C, once again resulted in larger Rr. Nonetheless, as seen in the inset of Fig. 3(c), at TDA < 650 °C, Rr was reduced by at least 5x when EIMP smaller than 80 keV was used. More importantly, the sample with EIMP = 45 keV & TDA = 575 °C exhibited a near-zero Rr, which can be ascribed to improved coupling between the superconducting Ga clusters.

Increasing the fluence during the implantation could further improve the connectivity of the Ga clusters. In Fig. 4(a), we show the TRIM simulations of implanted Ga concentration vs depth for EIMP = 45 keV and increasing ΦGa from 4 × 10^15 cm^-2 to 6 × 10^16 cm^-2. Increasing ΦGa only raises the peak height while it leaves the peak position unchanged, thereby increasing the Ga concentration per unit length within a narrow implanted Si region. Next, we prepared additional Si:Ga wafers with EIMP = 45 keV and two fluence levels: ΦGa = 5 × 10^15 cm^-2 and 6 × 10^16 cm^-2. Fig. 4(b) illustrates the Tc vs TDA for superconducting Si:Ga specimens with ΦGa = 4 – 6 × 10^16 cm^-2. Raising ΦGa significantly shifts the TDA window for superconductivity to lower temperatures. The shift is particularly noticeable for ΦGa = 6 × 10^16 cm^-2 where annealing at temperatures as low as 400 °C has led to superconductivity. Additionally, majority of the Tc values at that fluence fell below β-Ga Tc, which may be a result of excess disorder. At high fluence, Si and O recoil events are more frequent throughout the implantation process leading to higher disorder in the superconducting phase. In Fig. 4(c), we show the reentrant resistance Rr for the superconducting Si:Ga vs TDA and ΦGa. We notice that increasing ΦGa to 6 × 10^16 cm^-2 significantly reduces the dependence of Rr on the TDA while the absolute Rr values at 1.5 K remain below 0.1 Ω/□.

Figure 3. (a) TRIM simulations of Ga concentration vs depth at various implantation energies EIMP from 25 keV to 80 keV with a fixed fluence ΦGa of 4 × 10^15 cm^-2. In all simulations, a 30 nm thick SiO2 is accounted for. (b) Transition temperatures Tc vs anneal temperature TDA for all the superconducting Si:Ga samples with EIMP between 35 keV and 80 keV. (c) Reentrant resistance at 1.5 K Rr as a function of TDA and EIMP. The inset magnifies the region outlined by the box near Rr ~ 0 Ω/□.

Figure 4. (a) TRIM simulations of Ga concentration vs depth at fixed implantation energy EIMP of 45 keV when Ga+ fluence ΦGa varies between (4 × 10^15 cm^-2 and 6 × 10^16 cm^-2). In all simulations, a 30 nm thick SiO2 is accounted for. (b) Transition temperatures Tc vs anneal temperature TDA for all the superconducting Si:Ga samples with ΦGa = (4 × 10^15 cm^-2, 5 × 10^16 cm^-2, and 6 × 10^16 cm^-2). (c) Reentrant resistances at 1.5 K Rr vs TDA and ΦGa. The inset magnifies the region outlined by the box near Rr ~ 0 Ω/□.

Next, we turn to resistance measurements below 1 K car-
ried out in a Triton dilution refrigerator (Oxford Instruments). Fig 5 displays the magnitude of AC impedance $Z$ vs temperature from 20 mK to 1 K for three samples with processing conditions that include: (A) E$_{IMP}$ = 80 keV, $\Phi_{Ga}$ = 4 × 10$^{16}$ cm$^{-2}$, and $T_{DA}$ = 575 °C; (B) E$_{IMP}$ = 45 keV, $\Phi_{Ga}$ = 4 × 10$^{16}$ cm$^{-2}$, and $T_{DA}$ = 575 °C; (C) E$_{IMP}$ = 45 keV, $\Phi_{Ga}$ = 6 × 10$^{16}$ cm$^{-2}$, and $T_{DA}$ = 500 °C. Samples (B) and (C) were chosen because they showed the lowest $R_C$ at 1.5 K among all the samples presented in this study. In contrary, sample (A) represents a film with a clear reentrant behavior, although the smallest for samples with $E_{IMP}$ = 80 keV. The reason for using $Z$ instead of $R_C$ is the behavior of sample (A) below 1 K; the real part of $Z$ becomes negative while the imaginary part grows, consistent with an increasingly capacitive response. The $Z$ for (A) eventually reaches values as high as 20 $\Omega$ at 20 mK; about 29% of its normal impedance ($Z_{10K}$). In contrast, $Z$ for (B) and (C) are weakly temperature-dependent with negligible resistance down to 20 mK (here $Z \approx R$).

The TEM images and EDS maps displayed in Fig 5(b) & (c) help connect the low-temperature behavior of samples (B) & (C) to their structural characteristics, particularly their Ga cluster distributions. As a result of implantation at 45 keV, in both samples the depths of the disturbed regions have been reduced to 65 – 70 nm. For sample (B) in shown Fig 5(b) Ga clusters appear to form a 8 – 10 nm thick densely-packed layer with little discontinuity along the Si/SiO$_2$ interface. For sample (C) shown in Fig 5 which has lower $T_{DA}$ but higher fluence, Ga clusters are instead dispersed within the top half of the disturbed zone alongside a Ga-rich bands that formed 25 nm below the SiO$_2$ cap. Despite no clear evidence for continuity of Ga cluster networks, absence of reentrant behavior in sample (C) confirms a stronger coherent coupling between the clusters. This could be partially explained by 1.5x higher dose of Ga as dopant inside the Si weak-links effectively turning them into metals with no carrier freeze-out. Furthermore, the higher residual resistance for sample (C) relative to (B) may also be attributed to this difference in Ga cluster coupling mechanism.

Table I. Summary of critical superconducting parameters for the three samples whose reentrant resistances were measured down to 20 mK. CC limit represents the Clogston–Chandrasekhar limit for the upper critical field defined in Ref [20].

| #  | $E_{IMP}$ (keV) | $\Phi_{Ga}$ (cm$^{-2}$) | $T_{DA}$ (°C) | $T_c$ (T) | $B_0$ (T) | CC Limit (nm) | $\xi_0$ |
|----|----------------|--------------------------|--------------|---------|--------|--------------|--------|
| (A)| 80 keV         | 4 × 10$^{16}$            | 575          | 6.6     | 10.53  | 11.9         | 5.59   |
| (B)| 45 keV         | 4 × 10$^{16}$            | 575          | 7.0     | 12.06  | 12.6         | 5.22   |
| (C)| 45 keV         | 6 × 10$^{16}$            | 500          | 5.71    | 13.55  | 10.3         | 4.93   |

Finally, to further evaluate the superconducting phases observed in Si:Ga samples, we studied their resistive transitions in magnetic fields. Table I lists $T_c$, critical magnetic field ($B_0$) and coherence length ($\xi_0$) for samples with processing conditions identical to (A), (B), and (C) in Fig 5(a). Additionally, the Clogston–Chandrasekhar (CC) limit is listed on the table for each sample. All samples exhibit large $B_0$’s consistent with filamentary type-II nature of superconductivity for which the CC limit was developed. In sample (C), however, the CC limit is surpassed by about 3.25 T. Moderate levels of deviation from CC limit in very thin lead films has been attributed to strong spin-orbit coupling in the 2D metal [21]. Further studies will be underway to determine the possible roles of spin-orbit coupling and structural disorder in observation of such high critical fields.
In conclusion, we have demonstrated the conditions to maintain zero resistance in hyperdoped Si:Ga down to millikelvin temperatures. We first found a reentrant resistive transition below $T_c$ for samples prepared at higher implantation energies due to weak coupling between the superconducting Ga clusters. By monitoring the reentrant resistance over a wide parameter space of $E_{\text{IMP}}$ and $\Phi_G$, we targeted conditions that significantly improve the coherent coupling of Ga cluster, therefore, eliminating the reentrant transition at temperatures as low as 20 mK. Our results should open a path for integration of hyperdoped Si:Ga into gate-tunable Josephson junctions as basic building blocks for functional superconducting circuits.

We provide supplementary material that includes the optical images of Si:Ga surfaces, detailed resistance vs temperature traces, complementary TEM images and EDS line-scans of samples with 45 keV implantation energy, the magnetoresistance measurements, and the details of critical field and superconducting coherence length calculations.

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