Hard Superconducting Gap and Diffusion-Induced Superconductors in Ge–Si Nanowires

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Supporting Information

ABSTRACT: We show a hard superconducting gap in a Ge–Si nanowire Josephson transistor up to in-plane magnetic fields of 250 mT, an important step toward creating and detecting Majorana zero modes in this system. A hard gap requires a highly homogeneous tunneling heterointerface between the superconducting contacts and the semiconducting nanowire. This is realized by annealing devices at 180 °C during which aluminum interdiffuses and replaces the germanium in a section of the nanowire. Next to Al, we find a superconductor with lower critical temperature ($T_C = 0.9$ K) and a higher critical field ($B_C = 0.9$–1.2 T). We can therefore selectively switch either superconductor to the normal state by tuning the temperature and the magnetic field and observe that the additional superconductor induces a proximity supercurrent in the semiconducting part of the nanowire even when the Al is in the normal state. In another device where the diffusion of Al rendered the nanowire completely metallic, a superconductor with a much higher critical temperature ($T_C = 2.9$ K) and critical field ($B_C = 3.4$ T) is found. The small size of these diffusion-induced superconductors inside nanowires may be of special interest for applications requiring high magnetic fields in arbitrary direction.

KEYWORDS: Superconductor–semiconductor hybrid device, topological superconductivity, Majorana quasiparticle, Ge–Si nanowire, Josephson junction, hard superconducting gap

The discovery that Majorana fermions offer a route toward an inherently topologically protected fault-tolerant quantum computer marked the beginning of a quickly growing field of research to achieve their experimental realization. Majorana fermions require a topological superconducting material, which in practice can be realized by coupling a conventional $s$-wave superconductor to a one-dimensional nanowire with high spin–orbit coupling and $g$-factor. Signatures of Majorana fermions are expected to arise as a conductance peak at zero bias and finite magnetic fields. The first reports showing these zero-bias conductance peaks in InAs and InSb nanowires suffered from sizable subgap conductivity attributed to inhomogeneities in the nanowire–superconductor interface. The resulting quasiparticle poisoning decoheres Majorana states since they will participate in braiding operations and additionally obscure the Majorana signatures at zero energy. Strong efforts have been made to improve these interfaces, that is, induce a hard gap, using epitaxially grown Al or specialized surface treatments methods resulting in much better resolved Majorana signatures.

In contrast to the group III–V materials used in most previous work, we use Ge–Si core–shell nanowires consisting of a monocrystalline Ge core with a diameter of ~15 nm and a Si shell thickness of 2.5 nm covered by a native SiO$_2$. Coherent strain in the defect-free crystal structure results in high hole-mobilities. The electronic properties of the one-dimensional hole gas localized in the Ge core make them a promising candidate for observing Majorana fermions, although their interaction with a superconductor is still relatively unexplored. These wires are predicted to have a strong first-order Rashba type spin–orbit coupling which, together with the $g$-factor, is tunable by electric fields. Our devices consist of a nanowire channel with superconducting Al source and drain placed on an oxidized Si substrate (for more detailed information about the fabrication process see Supporting Information). We focus on two devices where an essential thermal annealing process results in interdiffusion between Al in the contacts and Ge in the nanowire channel. Device A is an electric-field tunable Josephson junction as shown in Figure 1a, whereas in device B the whole...
semiconducting nanowire channel has been metalized and we suspect Al has largely replaced the semiconductor.

The electric field dependence of Device A has already been extensively studied in ref 34 where the main result was the observation of two distinct regimes: a highly transparent regime with a near ideal $I_{\text{F}}R_{\text{S}}$ product in accumulation, and a tunneling regime with few-hole occupancy where supercurrent only appears at the charge degeneracy points. In this work, we extend on this by investigating the magnetic field dependence of the transport properties in both regimes.

To gain insight into the microscopic properties of the superconductor–semiconductor interfaces, we start by investigating Device A using high-angle annular dark-field–scanning transmission electron microscopy (HAADF-STEM) in combination with energy-dispersive X-ray spectroscopy (EDX). We find strong indications that the additional superconductor, as well as the highly homogeneous superconductor–nanowire interface arises during the thermal annealing process where Al interdiffuses with the material in the semiconducting nanowire. In the second part, we map the switching current $I_{\text{SW}}$ as a function of critical field $B_{C}$ and critical temperature $T_{C}$ of device A and B, which clearly shows an additional superconducting phase in both devices. In the final part, we investigate the hardness of the superconducting gap in the semiconducting nanowire of device A, by means of electronic transport measurements near depletion,20,23 and observe that the conductance in the gap is suppressed by a factor $\sim 1000$.

**Al–Ge Interdiffusion.** To investigate the effects of the annealing on the stoichiometric composition of the nanowire channel, a TEM lamella was made along the nanowire axes of device A as indicated in Figure 1a. We first apply a stack of protective SiO$_2$ and Pt layers and subsequently create the TEM lamella using a standard focused ion beam lift-out protocol. This allows us to perform an analysis on the cross-section of the device, as can be seen in Figure 1b. In both panels a and b in Figure 1, a smaller region (Area 1) with higher contrast on the left and a bigger region with lower contrast on the right (Area 2) can be observed. Figure 1c shows the resulting EDX signals in these regions for the elements Ge, Si, and Al, and we observe the following clear distinction: in Area 1 we observe a strong Ge signal whereas in Area 2 the signal is dominated by Al.

In Figure 1d, we show the integrated EDX spectra for both areas. When comparing the two areas, we observe that in Area 2 the Ge $L\alpha$, Ge $L\beta$, and Ge $K\alpha$ signals fall below the detection limit. As is the convention in EDX analysis, L and K denote the orbital to which an electron decays in a picture where K, L, and M are the outer atomic orbitals, whereas $\alpha$ and $\beta$ indicate whether it decays from the first or second higher orbital. The Al $K\alpha$ signal shows the opposite behavior, implying that Ge has been replaced by Al in Area 2. The counts for elements O, C, and Si remain equal in both areas (see also Figure S1). As we will discuss in the following section, the superconductor in Area 2 has profoundly different properties from the Al contacts and we therefore refer to it as X1. Interdiffusion has also taken place below the left contact without reaching the channel, although this is not evident from the TEM data. Instead, we conclude this from transport data in the next section (Figure 2 and Figure S3). As a side-note, we cannot observe the effects of the interdiffusion process on the Si shell, because the Si signal is dominated by the SiO$_2$ that covers the substrate.

An in-depth study on the thermally induced interdiffusion process between Al and pure Ge (111) nanowires, a highly similar system to ours, has been performed in refs 40 and 41. Here, in situ monitoring of the metal front inside the nanowires at various temperatures reveals that the velocity of propagation as a function of the length of the metalized nanowire segment is volume-diffusion limited and possibly surface-diffusion limited with the Al forming a monocrystalline face-centered cubic crystal inside the nanowire. The metal front forms an atomically sharp interface and no intermetallic phase is found in the metalized nanowire segment, that is, the Ge is transported out of the wire into the Al contacts. These observations are explained by a 15 orders of magnitude lower diffusion constant for Al in Ge than for Ge in Al.42,43 Furthermore, the initial start of the diffusion reaction is governed by the respective activation energies (121.3 kJ/mol for Ge in Al, 332.8 kJ/mol Al in Ge42,43) and may depend on the specific atomic arrangement of the initial nanowire–Al interface, explaining the variation in the starting time of the diffusion reaction, even for two separate contacts on the same wire. These findings largely correspond to our observations on Ge–Si core–shell nanowires and give an explanation for the asymmetry in our contacts (see Figure S2 for SEM images of...
partly and fully metalized nanowires), as well as the variation in device properties.

Two Superconductors in a Nanowire Josephson Junction. In Figure 2a, we show a magneto-spectroscopy of device A, the Josephson junction; we plot the differential resistance $\partial V_{SD}/\partial I_S$ versus the sourced current $I_S$ and the out-of-plane magnetic field $B_\perp$ (see illustration in Figure 3b) while sweeping $I_R$ from negative to positive current. The backgate $V_{BG}$ is fixed at $-4.7$ V where multiple subbands contribute to transport and the junction is highly transparent.

The superconducting region (black) is bounded by $I_R < I_S < I_{SW}$ with $I_R$ the retracking current at negative bias and $I_{SW}$ the switching current at positive bias. Upon increasing $B_\perp$ from 0, $I_{SW}$ decreases gradually until aluminum becomes normal at the critical out-of-plane field $B_{C,AL} \approx 40$ mT after which a finite $I_{SW}$ remains. For all $B_\perp$, $I_{SW} > I_R$, indicating that our junction is hysteretic for this particular value of $V_{BG}$ due to the junction being underdamped while additional heating-induced hysteresis cannot be excluded (see Figure S3a for a gate-dependence of $I_{SW}$ and $I_R$).

When increasing $B_\perp$ further in Figure 3b, $I_{SW}$ slowly decreases and finally disappears. The proximity-induced supercurrent above $B_{C,AL}$ implies the presence of a second superconducting material, X1, in or near the nanowire channel with a critical field $B_{C,X1} \approx 950$ mT. To confirm that our Al contacts are normal for $B_\perp > B_{C,AL}$, we consider the background resistance $R_B$ in the superconducting region as a function of $B_\perp$ in the bottom panel of Figure 2b. $R_B = 0$ for $B_\perp < B_{C,AL}$ whereas for $B_\perp > B_{C,AL}$ the background resistance gradually increases to $R_B \approx 0.25$ kΩ attributed to a normal series resistance of the Al contacts. Additionally, the out-of-plane critical field of a separately measured Al lead matches $B_{C,AL}$ (see Figure S4).

In Figure 2c, we show a magneto-spectroscopy at 900 mK and observe that X1 is quenched for all $B_\perp$, while Al still induces a supercurrent for $B_\perp < 1.25 kT$. This shows that X1 has a lower $T_C$ and a higher $B_C$ than the Al contacts. Because X1 has a higher $B_C$ and a lower $T_C$ than Al, we can selectively switch either superconductor to the normal state, resulting in four possible device configurations I–IV as illustrated in Figure 2 and summarized in the inset in Figure 2b (a precise set of conditions for each configuration can be found in Table S1). Figure 2d shows plots of $V_{SD}$ versus $I_S$ in all four configurations, clearly showing a supercurrent in configuration II where Al is normal and only X1 is superconducting. Because we observe a gate-tunable Josephson current even in configuration II, we conclude X1 is present on both sides of the Ge–Si segment (see Figure S3 for differential resistance maps versus backgate in all four configurations).

Junction $I_{SW}$ versus $B$ and $T$. For the observed superconductors and their specific geometries, the critical field and critical temperature are interdependent variables and...
may have a nontrivial relation; the boundaries of the configurations I–IV in terms of $B_C$ and $T_C$ cannot directly be deduced from the data in Figure 2. We therefore collect $I_{SW}$ versus $B$ from magneto-spectroscopies for a large number of temperatures and the three main magnetic field axes $B_\parallel$, $B_\perp$, and $B_\perp$ which are illustrated by the inset in Figure 3b. For the in-plane field perpendicular to the nanowire, $I_{SW}$ has two clearly distinct overlapping shapes as a function of $T$ and $B_\parallel$ in

**Table 1. Maximum values for $T_C$, $B_C$ of Al, X1, and X2 As Determined in Figure 3**

| Material | $T_C$ (K) | $\Delta$ (μV) | $B_C$ ★ (mT) | $B_C$ ⊥ (mT) | $B_C$ || (mT) |
|----------|----------|----------------|--------------|--------------|--------|
| Al       | 1.4 ± 0.05 | 212 ± 6       | 293 ± 10     | 41 ± 2       | 282 ± 10 |
| X1       | 0.9 ± 0.05 | 133 ± 8       | 1230 ± 10    | 909 ± 11     | 1010 ± 20 |
| X2       | 2.9 ± 0.1  | 441 ± 14      | 3.4 ± 0.1    |              |         |

*We take $T_{CAI}$ ($B_C = 0$), $T_{CX1}$ ($B_\perp = 50$ mT), and $B_C$ ($T \approx 0$) to obtain their respective maximum values. The BCS superconducting gap is determined as $\Delta = 1.764k_B T_C^4$.45*
Figure 3a. The "peak" extending to $T \approx 1400$ mK at $B = 0$ with a width of $|B_\perp| \approx 250$ mT at $T = 50$ mK is attributed to the superconducting state of Al, whereas the second shape (the "tail"), extending up to $\sim 1000$ mT at $T = 50$ mK, corresponds to the superconducting phase of X1. We can thus map the four configurations in the color plot on the $T$ versus $B_\parallel$ axes. We now extract both the $T_C$-$B_{C,*}$Al and $T_C$-$B_{C,*}$X1 curves from Figure 3a (see Supporting Information Section SIII), that is, the critical temperature–critical field relation for Al and X1, and plot them in Figure 3b. We perform the same procedure for field directions $B_\perp$ and $B_\parallel$ (see Figure S5 for $I_{SW}$ versus $T$ and $B_\parallel$ and $B_\perp$).

Figure 4. Hard superconducting gap in a Ge–Si nanowire Josephson FET (Device A). (a) Differential conductance $\partial I_D/\partial V_{SD}$ versus $V_{SD}$ and $V_{BG}$. Odd (O) and even (E) hole occupation are denoted. The first two MAR orders are indicated at $V_{SD} = 2\Delta_{Al}$ and $\Delta_{Al}$. (b) Vertical linecuts from (a) showing $\partial I_D/\partial V_{SD}$ versus $V_{SD}$ at 50 mV intervals in $V_{BG}$. Curves are offset by 0.2 $\mu$S. (c) Averaged in-gap conductance $\langle G_{G} \rangle$ (black) and outside-gap conductance $\langle G_{O} \rangle$ (blue) versus $V_{BG}$. Dashed curves show theoretical minimal values and are the result of plotting eq 1. For every $V_{BG}$, $\langle G_{G} \rangle$ and $\langle G_{O} \rangle$ are averaged over a range of $V_{SD}$ as indicated by the gray area in (b) and the gray dashed lines in Figure S7, respectively. (d) $\partial I_D/\partial V_{SD}$ versus $V_{SD}$ for $B_\parallel$ from 0 to 1000 mT at 50 mT intervals. Curves are offset by 0.3 $\mu$S. Dashed lines show the expected position of the quasiparticle peak for $2\Delta_{Al}$ ($2\Delta_{X1}$) at $B = 0$. (e) Ratio $\langle G_{G}/G_{O} \rangle$ for the three main field axes $B_\perp$, $B_\parallel$, and $B_\star$ at $V_{BG} = 4.45$ V (blue line in (a–c)). Ranges in $V_{SD}$ where $\langle G_{G} \rangle$ and $\langle G_{O} \rangle$ are extracted are shown as gray areas in (d).
In Table 1, we summarize the maximum $T_C$, the resulting superconducting gap $\Delta$, and $B_C$ in the three field directions for Al and X1. Comparing $B_{C,AL} = 41$ mT with $B_{C,X1} = 293$ mT and $B_{C,AL} = 282$ mT, we notice a factor $\sim 7$ difference. This strong anisotropy for the out-of-plane field direction is clearly present in the $T_{C,Al}$-$B_{C,Al}$ curves in Figure 3b and is expected for the large aspect ratio of the 50 nm thick Al contacts.

The $T_{C,X1}$-$B_{C,X1}$ curves show a less prominent magnetic field anisotropy from which we can roughly deduce the shape of X1 by assuming that the normal surface of the material is inversely proportional to the critical field, that is, a larger superconducting normal-surface requires expelling more flux. Using the respective ratios of $B_{C,X1}$, $B_{C,Al}$, and $B_{C,Al}$, we observe that X1 is slightly elongated along the nanowire axis, reaffirming the hypothesis that X1 resides in the nanowire channel.

We now switch to the completely metalized device B where we believe Al has diffused completely through the channel, effectively making the nanowire a metallic superconductor. Figure 3c shows $I_{SW}$ versus $T$ and $B_{LC}$ to which the corresponding $T_{C,X1}$-$B_{C,X1}$ relation in Figure 3d is obtained by the previously mentioned polynomial fitting method. We see a critical temperature $T_{C,X1}$ = 2.9 K at $B = 0$ and critical field $B_{C,X1}$ = 3.4 T at $T = 50$ mK, both much higher than for X1 and the Al contacts. The switching current $I_{SW}$ = 1.5 mA is 2 orders of magnitude higher compared to device A.

When comparing $T_{C,X1}$ = 2.9 K and $B_{C,X1}$ = 3.4 T with thin Al aluminum films, we observe X2 has equivalent properties of an ~3 nm thick film (in parallel field) and we could conclude that X2 is simply a very small cylinder of aluminum inside the nanowire channel. However, for X1 with $T_{C,X1}$ = 0.9 K and $B_{C,X1}$ = 1 T an equivalent film thickness cannot be defined. Even though no intermetallic phases were found for annealed pure Ge nanowires in refs 40 and 41, a possible origin of X1 is the formation of a Al-Si/Ge alloy in our core–shell nanowires, albeit with a ratio of semiconductor to Al below stoichiometric compositions indeed result in a lower conductance value inside (outside) the gap averaged over a range of $V_{SD}$ from 0.5 K up to 11 K by various methods.

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broadened quantum dot level (also see ref 34). However, the obtained theoretical minimal in-gap conductance should be considered an approximation because we do not take into account any difference in interface transparency between the two contacts.

When measured in a SNS configuration, the ratio \( \frac{G_{C}(\Delta)}{G_{C}(\Delta)} \) gives an upper limit and could in reality be lower because it can be increased due to several other reasons than quasiparticle poisoning. (1) For higher \( V_{BG} \), \( G_{C}(\Delta) \) is limited by the noise floor of our measurement setup and does not further decrease. The decrease of \( G_{C}(\Delta) \) now lowers the observed current suppression \( G_{C}(\Delta)/G_{C}(\Delta) \). (2) For lower \( V_{BG} \) MAR and the zero-bias peak, both characteristic for Josephson junctions, appear as conductance peaks inside the gap which leads to a decreased \( G_{C}(\Delta)/G_{C}(\Delta) \). (3) The quantum dot in the junction may lead to Fabry–Perot resonances and Kondo-enhanced tunnelling around zero bias (see Figure S6). SN devices will not exhibit these effects and may therefore result in a lower ratio \( G_{C}(\Delta)/G_{C}(\Delta) \) and give a better approximation of the quasiparticle density in the gap. Because of this, we cannot directly compare the current suppression in our device with other work probing the superconducting gap using a single superconducting contact. Nevertheless, the fact that our \( G_{C}(\Delta)/G_{C}(\Delta) \) is limited by the noise floor our measurement setup suggests that our semiconductor–nanowire interface homogeneity could be comparable to InAs nanowire devices using epitaxial growth techniques or specialized surface treatments.

We will now look at the magnetic field dependence of the hardness of the gap. We fix \( V_{BG} \) at 4.45 V and plot \( \partial G_{C}/\partial V_{SD} \) versus \( V_{SD} \) for several \( B_{x} \) in Figure 4d. For increasing \( B_{x} \), the sharp quasiparticle peak at \( V_{SD} = 2\Delta_{k} \) reduces in height and broadens up to \( B_{c3-}^{\perp} \approx 300 \) mT. Above \( B_{c3-}^{\perp} \) we enter configuration II where only X1 is superconducting but which fails to produce a clear second quasiparticle peak at \( \sim 2\Delta_{X1} \). Instead, we see a “soft gap” signature persisting up to \( B_{c3-}^{\perp} \), which we attribute to X1 having an ill-defined gap due to possible diffusion-induced spatial variations in its stoichiometry or geometry.

In Figure 4e, we plot the ratio \( \frac{G_{C}(\Delta)}{G_{C}(\Delta)} \) for the three main field directions. The initial ratio \( \sim 1 \times 10^{-3} \) in configuration I as defined in Figure 2 and the gap remains hard until we approach the critical field of AI for the respective field direction as summarized in Table 1 (see Figure S8 for the corresponding differential conductance maps for all three main field axes). The highest field where the gap remains hard, \( B_{c1} \approx 250 \) mT, is slightly lower than \( B_{c1}^{Al} \) because of the strongly reduced \( \Delta_{Al} \) at this field. The much softer gap in configuration II induced by X1 leads to a \( \frac{G_{C}(\Delta)}{G_{C}(\Delta)} \approx 1 \times 10^{-3} \) which gradually increases to 1 approaching \( B_{c2-3}^{\perp} \).

Another example of the change in transport properties when AI becomes normal is seen in Figure 2a,c. Here, the fringes in the normal state attributed to MAR are only visible for \( B_{c1}^{Al} \). For \( B_{c1}^{Al} \), the absence of MAR suggests an increase of inelastic processes due to an ill-defined induced gap or a greatly increased quasiparticle poisoning rate.

The results in Figure 4e show that the AI contacts needs to be superconducting in order to observe a hard gap. On the other hand, when only Al is superconducting, that is, going from configuration I to III, we observed no change in \( G_{C}(\Delta) \) that can be attributed to X1 becoming normal (see Figure S9 for the temperature dependence of the differential conductance at \( V_{BG} = 4.45 \) V and \( B = 0 \)). This suggests that X1 does not need to be a superconductor to observe a hard gap as long as the Al contacts proximate the entire junction. This is likely to happen, because the transparency between AI and X1 is high, and \( \Delta_{Al} > \Delta_{X1} \) indicating a coherence length for X1 comparable or larger than for Al, that is, in the order of micrometers.

Previously, in this system a soft gap signature using NbTiN contacts has been shown as well as a hard gap using Al contacts. This work adds an investigation of the superconductor–semiconductor interfaces and their microscopic properties. We therefore revisit Figure 1b,c and take a closer look at the interface between the X1 and the Ge–Si island. Even though our TEM and EDX resolution prohibits a conclusive statement about the interface properties on an atomic scale, the abrupt change in contrast suggests an upper limit for the interface width of a few nanometer. As explained, this observation is supported by refs 40 and 41 showing an atomically sharp interface between the Ge and Al segment where both remain crystalline. This type of interface would fit our observation of a hard gap, requiring a defect-free highly homogeneous heterointerface and low junction transparency close to depletion. This indicates that the interdiffusion reaction between Ge and Al is essential for the observed hard superconducting gap.

Utilizing these interfaces in devices suitable for measuring Majorana fermions in this system would require a high level of control over the interdiffusion process, that is, lateral diffusion and metalization of nanowire segments should be prevented. One route would be to perform device annealing while in situ monitoring of the diffusion process as in ref 41, or possibly a higher level of control could be achieved by optimizing the annealing process. In addition, one would require thinner Al leads in order to withstand the required in-plane magnetic fields (>1 T) to reach the topological phase transition.

With a controlled interdiffusion reaction, the superconductors X1 and X2 themselves would also pose as interesting materials, because their high \( B_{c} \) in relation to their superconducting gaps might allow the creation of Majorana fermions in materials where low g-factors could be limiting. However, more research is required to understand the soft gap induced by X1 and to fully explore the possible superconductors, their composition, and formation process.

In conclusion, we have shown that Ge–Si nanowire devices with Al contacts contain additional superconductors after annealing, caused by diffusion of Al into the nanowire channel. We identify two superconductors in two different devices: X1 is present in a Josephson FET and X2 resides in a metallic nanowire channel. Both X1 and X2 remain superconducting for magnetic fields much higher than the Al contacts which could be of potential interest for applications where proximity-induced superconductivity is required in high magnetic fields.

Close to depletion, the Josephson FET exhibits a hard superconducting gap where the in-gap conductance is suppressed by a factor \( \sim 1000 \) in an SNS configuration where the in-gap conductance is close to the approximate theoretical minimum. The gap remains hard up to magnetic fields of \( \sim 250 \) mT. For higher fields, a soft gap remains up to the critical field of X1. We can selectively switch Al or X1 from the normal to the superconducting state and, combined with the results of the TEM and EDX analysis, this leads us to believe that the diffusion-induced homogeneous heterointerface between the Ge core and the metalized nanowire segment is key in obtaining this hard gap. The next challenge is to more precisely
control the diffusion of Al which would grant a highly promising system for observing Majorana zero modes.30

■ ASSOCIATED CONTENT

Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.nanolett.9b03438.

Fabrication details, specific methods used for data analysis, and additional figures (PDF)

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Notes
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